# IN THE UNITED STATES DISTRICT COURT FOR THE DISTRICT OF MINNESOTA

# OXYGENATOR WATER TECHNOLOGIES, INC.

Plaintiff,

v.

Civil Action No. 0:20-cv-00358 (ECT/HB)

TENNANT COMPANY

Defendant.

# JOINT APPENDIX

# Table of Contents for Joint AppendixOxygenator Water Technologies, Inc. v. Tennant CompanyC.A. No. 20-cv-0358

Description	Appendix Page No.
U.S. Reissued Patent No. RE45,415 E	JA1
U.S. Reissued Patent No. RE47,092 E	JA17
U.S. Reissued Patent No. RE47,665 E	JA38
U.S. Patent No. 6,689,262	JA58
U.S. Patent No. 7,396,441	JA67
U.S. Patent No. 7,670,495	JA83
File History of U.S. Patent No. 6,689,262	JA100
File History of U.S. Patent No. 7,396,441	JA321
File History of U.S. Patent No. 7,670,495	JA660
File History of U.S. Reissued Patent No. RE45,415 E	JA981
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CASE 0:20-cv-00358-ECT-HB Docu



# (19) **United States**

# (12) Reissued Patent Senkiw

(10) Patent Number:USRE45,415 E(45) Date of Reissued Patent:Mar. 17, 2015

### (54) FLOW-THROUGH OXYGENATOR

- (75) Inventor: James Andrew Senkiw, Minneapolis, MN (US)
- (73) Assignee: Oxygenator Water Technologies, Inc., St. Louis Park, MN (US)
- (21) Appl. No.: 13/247,241
- (22) Filed: Sep. 28, 2011

#### **Related U.S. Patent Documents**

Reissue of:

(64)

uc 01.	
Patent No.:	7,670,495
Issued:	Mar. 2, 2010
Appl. No.:	12/023,431
Filed:	Jan. 31, 2008

- U.S. Applications:
- (60) Division of application No. 10/732,326, filed on Dec. 10, 2003, now Pat. No. 7,396,441, which is a continuation-in-part of application No. 10/372,017, filed on Feb. 21, 2003, now Pat. No. 6,689,262.
- (60) Provisional application No. 60/358,534, filed on Feb. 22, 2002.
- (51) Int. Cl. *C02F 1/48* (2006.01) *C02F 1/00* (2006.01)

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- (52) U.S. Cl. USPC ...... 210/739; 204/157.15; 204/245; 204/232; 204/628; 204/600; 210/600; 210/243; 210/153; 422/22; 422/186; 422/186.04
- (58) Field of Classification Search

205/701, 628, 633, 742, 756, 757; 22/192, 321.7, 1; 119/263 See application file for complete search history.

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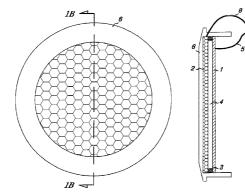
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# (57) ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

#### 16 Claims, 8 Drawing Sheets



CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 3 of 17

# US RE45,415 E

Page 2

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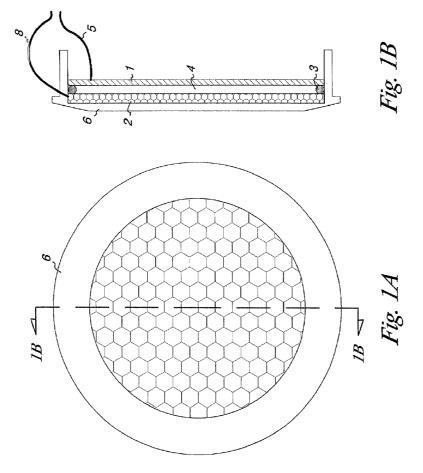
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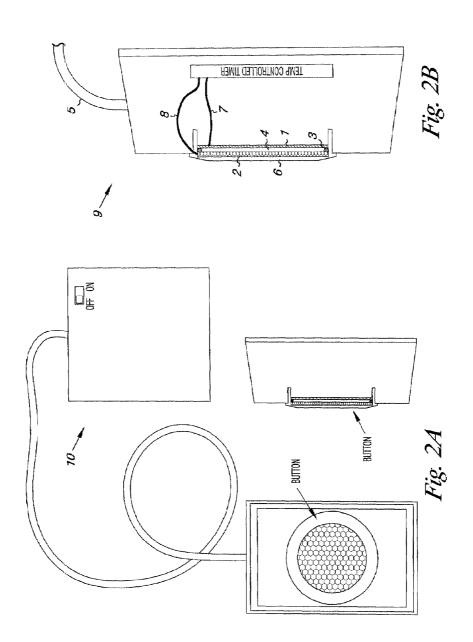
CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 4 of 17

U.S. Patent Mar. 17, 2015 Sheet 1 of 8 US RE45,415 E



CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 5 of 17

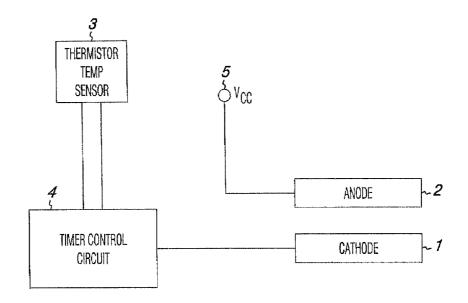
U.S. Patent Mar. 17, 2015 Sheet 2 of 8 US RE45,415 E



# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 7 of 1320

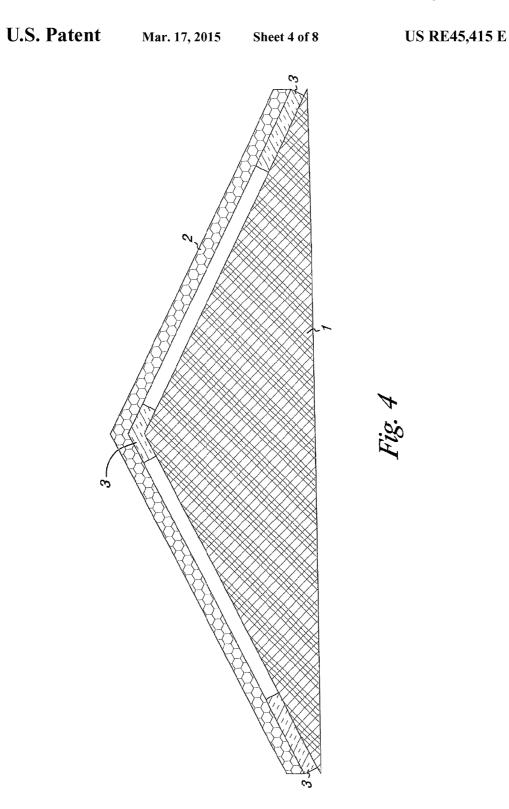
CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 6 of 17

U.S. Patent Mar. 17, 2015 Sheet 3 of 8 US RE45,415 E



*Fig. 3* 

CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 7 of 17



# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 9 of 1320

CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 8 of 17

**U.S.** Patent

Mar. 17, 2015

Sheet 5 of 8

US RE45,415 E

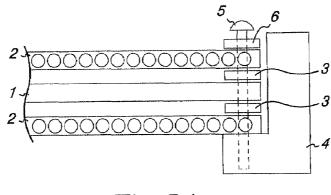
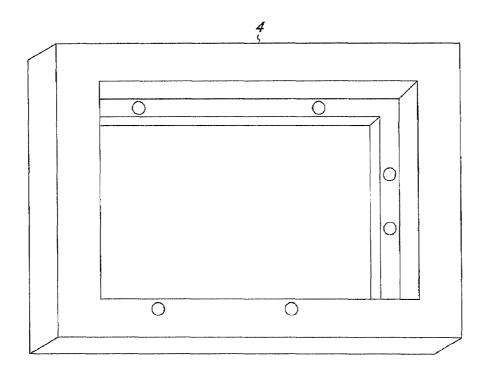


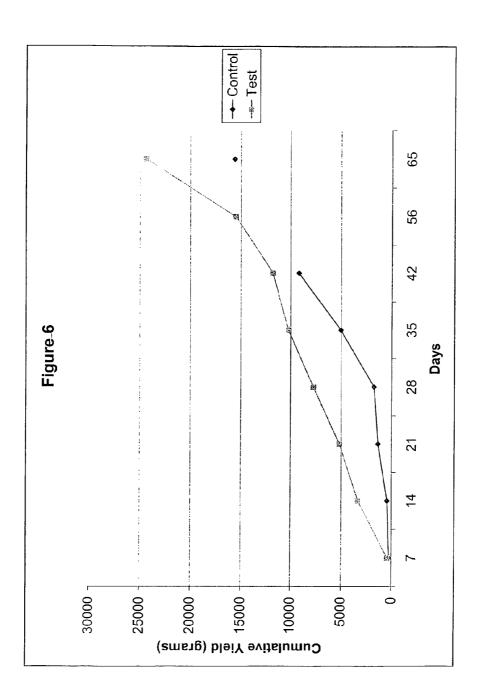
Fig. 5A



*Fig.* 5*B* 

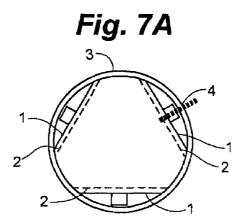
CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 9 of 17

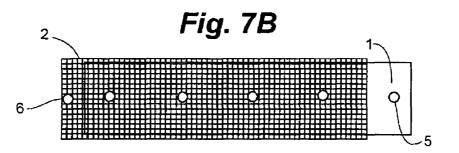
U.S. Patent Mar. 17, 2015 Sheet 6 of 8 US RE45,415 E



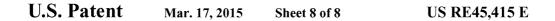
CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 10 of 17

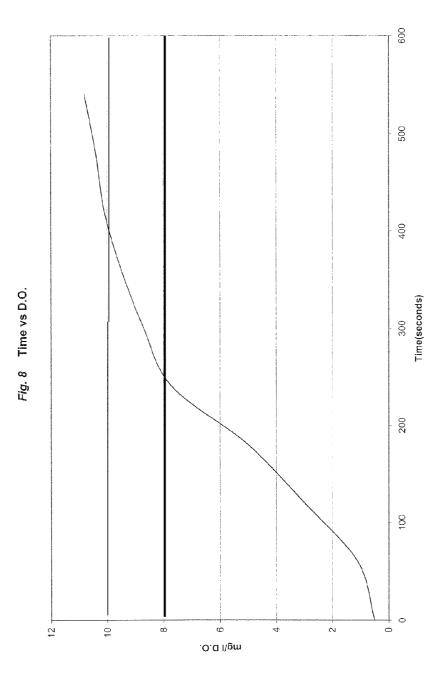
U.S. Patent Mar. 17, 2015 Sheet 7 of 8 US RE45,415 E





CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 11 of 17





OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 12 of 17

## US RE45,415 E

#### 1 FLOW-THROUGH OXYGENATOR

Matter enclosed in heavy brackets [] appears in the original patent but forms no part of this reissue specification; matter printed in italics indicates the additions made by reissue; a claim printed with strikethrough indicates that the claim was canceled, disclaimed, or held invalid by a prior post-patent action or proceeding.

## RELATED APPLICATIONS

This application is a division of application Ser. No. 10/732,326 filed Dec. 10, 2003 *now U.S. Pat. No. 7,396,441*, which in turn is a continuation-in-part of application Ser. No. <sup>15</sup> 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve 35 higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish 4 held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit 45 from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated 50 by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for 55 fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534, 143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diam-65 eter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 2

to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$
NET REACTION:	$6\mathrm{H}_{2}\mathrm{O} \rightarrow 4\mathrm{OH}^{-} + 4\mathrm{H}^{+} + + 2\mathrm{H}_{2} + \mathrm{O}_{2}$

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these 25 corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 13 of 17

## US RE45,415 E

3

and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a {fraction ( $V_{10}$ }) inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The pre-<sup>15</sup> ferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown 40 in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the  $O_2$  emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the  $O_2$  emitter.

FIG. 4 shows a funnel or pyramid variation of the  $\mathrm{O_2}^{-50}$  emitter.

FIG. 5 shows a multilayer sandwich  $O_2$  emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-55 through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.  $^{\,\,60}$ 

## DETAILED DESCRIPTION OF THE INVENTION

#### Definitions

For the purpose of describing the present invention, the following terms have these meanings:

4

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

" $O_2$  emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button <sup>45</sup> emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to <sup>50</sup> be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

## EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen. The anode and 65 cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 14 of 17

## US RE45,415 E

connection point 5. FIG. 2 shows a plan view of the assembled device. The  $O_2$  emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of 5 sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon®, polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.  $_{20}$ 

5

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain <sup>30</sup> duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. **3** shows a block diagram of a timer sor **3**, timer control circuit **4** and wire from a direct current power source **5**.

#### EXAMPLE 2

#### Measurement of O2 Bubbles

Attempts were made to measure the diameter of the  $O_2$ bubbles emitted by the device of Example 1. In the case of 45 particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, 50 backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was 55 enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the 60 scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so 65 monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to

6

increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>a</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treat-25 ing large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery  $_{40}$  supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

TABLE I

Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts
Bait keeper	5	6	0.090	0.060	0.36
Livewell	32	12	0.180	0.120	1.44
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5.000	2.500	30.00

### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An  $O_2$  emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodi-

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 15 of 17

## US RE45,415 E

7 ment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

cathode screen	0.045 inches thick
nylon spacer	0.053 inches thick
anode grid	0.035 inches thick
nylon spacer	0.053 inches thick
cathode screen	0.045 inches thick

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

#### EXAMPLE 5

# Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the  $_{40}$ web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with 45 oxygen, but it is unlikely that the water is superoxygenated. A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2  $\frac{1}{2}$  gallon water reservoir for the Control plant was 50eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked 60 like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three 65 months. At that time, the Test plant but not the Control plant had blossomed.

8

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in oneinch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least 20 eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

TABI	Æ	Π

Week of:	Control, gra tomatoes from plants/cumulati	eight	Test, grams tomatoes from seven plants/cumulative total		
July 27	240		400		
August 3	180	420	2910	3310	
August 10	905	1325	1830	5140	
August 17	410	1735	2590	7730	
August 24	3300	5035	2470	10200	
August 31	4150	9175	1580	11780	
September 15	not weighed		3710	15490	
Final Harvest September 24	6435	15620	8895	24385	

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. **6** shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 16 of 17

## US RE45,415 E

9

experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

#### EXAMPLE 6

#### Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently 10 was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120° angles to each other. The anodes and cathodes are positioned with 1 stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing  $_{20}$ hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other 25 embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and 30 flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was  $12.2^{\circ}$  C. but the flowing water cooled slightly to 11 or  $11.5^{\circ}$  C. Without undue experimentation, anyone may easily select the embodiment that best 35 suits desired characteristics from Table III or designed with the teachings of Table III.

10

It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only 5 once a day.

#### EXAMPLE 7

#### Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

The invention claimed is:

1. A method for treating waste water comprising;

providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other,

placing the emitter within a conduit; and

passing waste water through the conduit.

2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between

MODEL	ACTIVE ELECTRODE AREA, SQ. IN.	VOLTAGE	CURRENT, AMPS.	FLOW RATE GAL/MINUTE	DO OF* SAMPLE AT ONE MINUTE
2-Inch "T"	2	28.3	0.72	12	N/A
3-inch "T"	3	28.3	1.75	12	N/A
2-plate Tube	20	28.3	9.1	12	8.4
3-Plate tube	30	28.3	12.8	12	9.6

TABLE III

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The one-minute time point shows the rapid increase in oxygenation.

oxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The 55 conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irriga- 60 tion per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the 65 possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

The following plants will be tested for response to super- 50 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breaking the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

[3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.]

[4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.]

[5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.]

[6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.

# **OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

**JA15** 

# CASE 0:20-cv-00358-ECT-HB Document 9-1 Filed 05/08/20 Page 17 of 17

## US RE45,415 E

11

[7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.][8. A method for oxygenating a non-native habitat for

temporarily keeping aquatic animals, comprising:

inserting the emitter of claim **2** into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.]

**[9**. A method for lowering the biologic oxygen demand of polluted water comprising:

passing the polluted water through a vessel containing the <sup>10</sup> emitter of claim **2**.]

[10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.]

[11. The emitter of claim 2, further comprising a timer <sup>15</sup> control.]

**[12**. The emitter of claim **2**, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.]

13. A method for producing an oxygenated aqueous composition comprising:

flowing water at a flow rate no greater than 12 gallons per minute through an electrolysis emitter comprising an electrical power source electrically connected to an anode electrode and a cathode electrode contained in a <sup>25</sup> tubular housing,

causing electricity to flow from the power source to the electrodes, and,

- producing the composition comprising a suspension comprising oxygen microbubbles and nanobubbles in the <sup>30</sup> water, the microbubbles and nanobubbles having a bubble diameter of less than 50 microns, wherein:
  - the anode electrode is separated at a critical distance from the cathode such that the critical distance is from 0.005 inches to 0.140 inches;
  - the power source produces a voltage no greater than about 28.3 volts and an amperage no greater than about 13 amps,
  - the tubular housing has an inlet and an outlet and a tubular flow axis from the inlet to the outlet;
- the water flows in the inlet, out the outlet, is in fluid connection with the electrodes, and the water flowing into the inlet has a conductivity produced by the presence of dissolved solids such that the water supports plant or animal life.

14. A method according to claim 13 wherein the housing contains at least one anode and at least one cathode, the

12

electrodes are of a grid or solid design and are relatively positioned in cross section along the radius of the tubular housing with their long axes substantially parallel to the tubular water flow axis of the housing.

15. A method according to claim 13 wherein the housing has a side arm positioned at an angle relative to the tubular flow axis and the electrodes are located in the side arm.

16. A method according to claim 15 wherein the side arm contains a multiple number of anode and cathode electrodes and the electrodes are plate shaped.

17. A method according to claim 14 wherein a multiple number of anode and cathodes are present and are of grid or solid design.

18. A method according to claim 13 wherein the water has a temperature no greater than about ambient temperature at the inlet and the water temperature is a factor for formation of the suspension.

19. A method according to claim 13 wherein the microbubbles and nanobubbles remain in the water at least in part for a period up to several hours.

20. A method according to claim 19 wherein the period for which the microbubbles and nanobubbles at least in part remain in the water is determined by containing the water with microbubbles and nanobubbles in a two and one half gallon aquarium reservoir container.

21. A method according to claim 13 wherein the microbubbles and nanobubbles supersaturate the water.

22. A method according to claim 13 wherein the bubble diameter of the microbubbles and nanobubbles is less than 0.0006 inches.

23. A method according to claim 13 wherein the separation of electrodes is maintained by a nonconductive spacer.

24. A method according to claim 13 wherein the electrode separation distance is about 0.045 to about 0.06 inches.

25. A method according to claim 13 wherein the microbubbles and nanobubbles are substantially incapable of breaking the surface tension of the water.

26. A method according to claim 13 wherein each anode and cathode electrode of the emitter is positioned so that 40 substantially all points midway between opposing anode and cathode electrodes are closer to a surface of the tubular housing than to a center point within the tubular housing.

27. A method according to claim 26 wherein each anode and cathode electrode of the emitter are positioned so that the 45 electrodes do not obstruct a water flow passage along the center of the tubular housing.

\* \* \* \* \*

CASE 0:20-cv-00358-ECT-HB Docu



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# (19) United States

# (12) **Reissued Patent** (10) Senkiw (45)

# (10)Patent Number:USRE47,092 E(45)Date of Reissued Patent:\*Oct. 23, 2018

## (54) FLOW-THROUGH OXYGENATOR

- (71) Applicant: Oxygenator Water Technologies, Inc., St. Louis Park, MN (US)
- (72) Inventor: James Andrew Senkiw, Minneapolis, MN (US)
- (\*) Notice: This patent is subject to a terminal disclaimer.
- (21) Appl. No.: 15/085,741
- (22) Filed: Mar. 30, 2016

**Related U.S. Patent Documents** 

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(64)	Patent No.:	7,670,495
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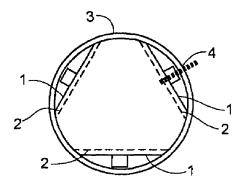
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Primary Examiner — Krisanne M Jastrzab (74) Attorney, Agent, or Firm — Carlson Caspers

#### (57) ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

#### 72 Claims, 8 Drawing Sheets



# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 3 of 22

# US RE47,092 E

Page 2

#### **Related U.S. Application Data**

No. 13/247,241, filed on Sep. 28, 2011, now Pat. No. Re. 45,415, which is a division of application No. 10/732,326, filed on Dec. 10, 2003, now Pat. No. 7,396,441, which is a continuation-in-part of application No. 10/372,017, filed on Feb. 21, 2003, now Pat. No. 6,689,262.

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CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 4 of 22

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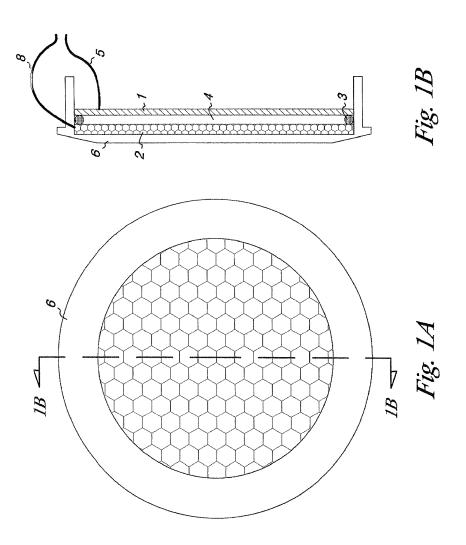
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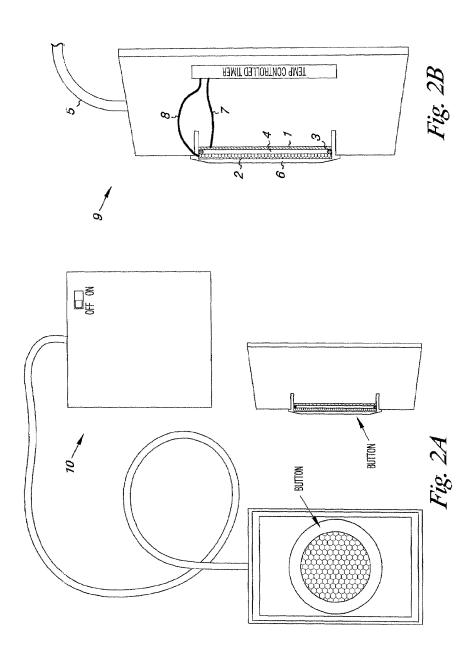
CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 5 of 22

U.S. Patent Oct. 23, 2018 Sheet 1 of 8 US RE47,092 E



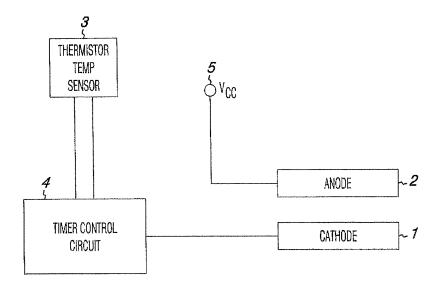
CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 6 of 22

U.S. Patent Oct. 23, 2018 Sheet 2 of 8 US RE47,092 E



CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 7 of 22

U.S. Patent Oct. 23, 2018 Sheet 3 of 8 US RE47,092 E



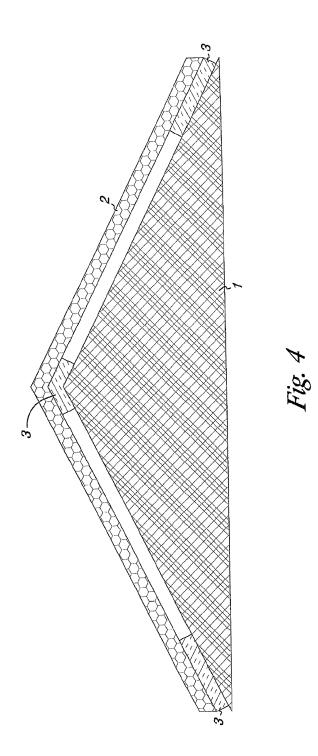
*Fig. 3* 

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA22

CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 8 of 22

U.S. Patent Oct. 23, 2018 Sheet 4 of 8 US RE47,092 E



# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 26 of 1320

CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 9 of 22

U.S. Patent Oct. 23, 2018 Sheet 5 of 8 US RE47,092 E

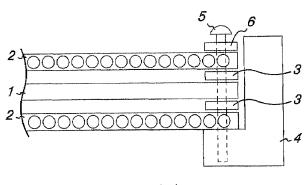


Fig. 5A

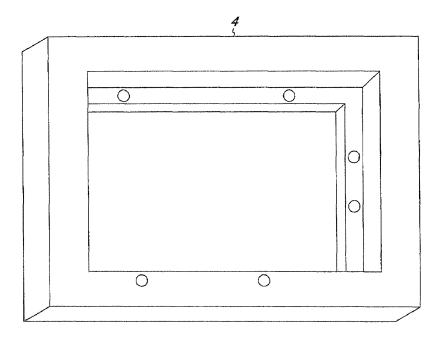
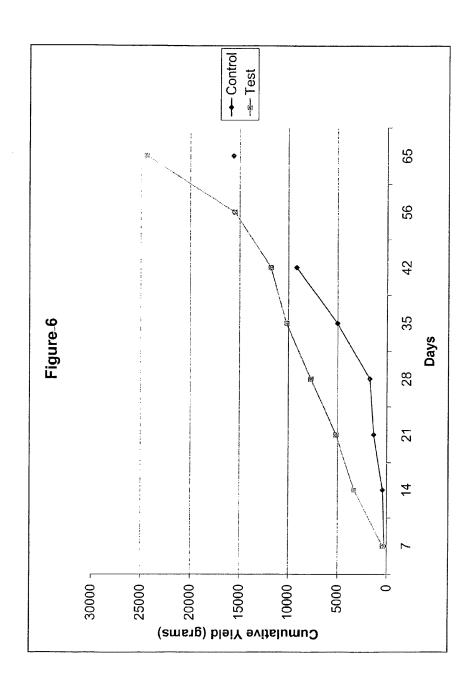


Fig. 5B

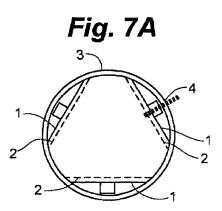
CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 10 of 22

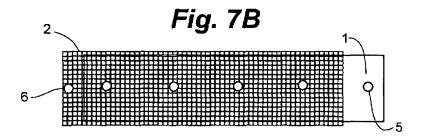
U.S. Patent Oct. 23, 2018 Sheet 6 of 8 US RE47,092 E



CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 11 of 22

U.S. Patent Oct. 23, 2018 Sheet 7 of 8 US RE47,092 E

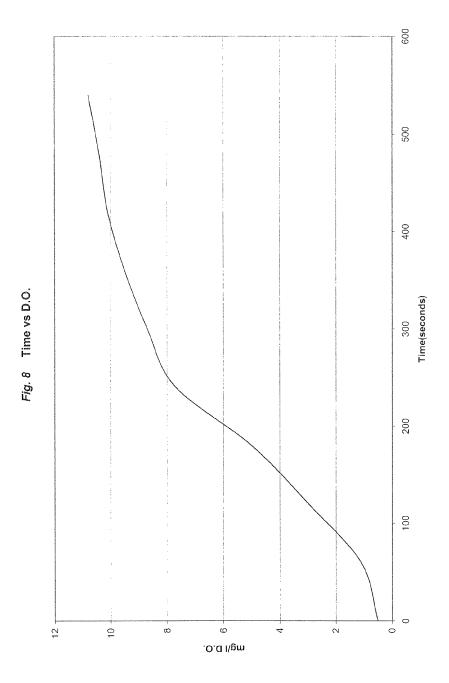




OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA26

CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 12 of 22



## CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 13 of 22

## US RE47,092 E

10

#### 1 FLOW-THROUGH OXYGENATOR

Matter enclosed in heavy brackets [] appears in the original patent but forms no part of this reissue specification; matter printed in italics indicates the additions made by reissue; a claim printed with strikethrough indicates that the claim was canceled, disclaimed, or held invalid by a prior post-patent action or proceeding.

#### RELATED APPLICATIONS

More than one reissue application has been filed for the reissue of U.S. Pat. No. 7,670,495. This application is a continuation reissue application of application Ser. No. 15 14/601,340, filed Jan. 21, 2015, which is a continuation reissue application of application Ser. No. 13/247,241, filed Sep. 28, 2011, now U.S. Pat. No. RE45,415, which is a reissue of U.S. Pat. No. 7,670,495. U.S. Pat. No. 6,760,495 is a division of application Ser. No. 10/732,326 filed Dec. 20 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Proviof which is hereby fully incorporated herein by reference. <sup>25</sup> 286 kilojoules of energy is required to generate one mole of

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content 30 of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to 40 achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For 45 example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the 50 respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of 55 these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to 60 make more oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into 65 the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by

2

increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

NET REACTION: $6H_2O \rightarrow 4OH^- + 4H^+ + 2H_2 + O_2$		AT THE CATHODE: AT THE ANODE: NET REACTION:	$\begin{array}{l} 4\mathrm{H}_{2}\mathrm{O}+4\mathrm{e}^{-}\rightarrow4\mathrm{OH}^{-}+2\mathrm{H}_{2}\\ 2\mathrm{H}_{2}\mathrm{O}\rightarrow\mathrm{O}_{2}+4\mathrm{H}^{+}+4\mathrm{e}^{-}\\ 6\mathrm{H}_{2}\mathrm{O}\rightarrow4\mathrm{OH}^{-}+4\mathrm{H}^{+}+2\mathrm{H}_{2}+\mathrm{O}_{2} \end{array}$
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oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 14 of 22

## US RE47,092 E

3

with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be <sup>5</sup> applied to field as well as hydroponic culture.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an <sup>10</sup> electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one <sup>15</sup> metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed <sup>20</sup> surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a {fraction ( $V_{16}$ }) inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the <sup>25</sup> anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The <sup>30</sup> public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held. Controls are provided to regulate the current and timing of <sup>35</sup> electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode 40 is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" 42 model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest. 50

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the <sup>55</sup> flow-through model for drip irrigation of crops and waste water treatment is disclosed.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the  $O_2$  emitter of the invention.

FIG. 2 is an assembled device.

FIG. **3** is a diagram of the electronic controls of the  $O_2$  emitter.

FIG. 4 shows a funnel or pyramid variation of the  $O_2$  65 emitter.

FIG. 5 shows a multilayer sandwich  $O_2$  emitter.

4

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flowthrough applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

# DETAILED DESCRIPTION OF THE INVENTION

#### Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

" $O_2$  emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At

**JA29** 

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 15 of 22

## US RE47,092 E

10

5

distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode **2** is a (fraction  $(\frac{1}{16})$ } inch mesh (size 8 mesh) marine stainless steel screen. The anode and 1 cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2 shows a plan view of the assembled device. The  $O_2$  emitter 6 with the anode connect- 20 ing wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no 25 more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most prefer- 30 ably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon®, polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a noncompressible spacer. It was found that Buna, with a durom- 35 eter measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical 40 distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises <sup>45</sup> a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given <sup>50</sup> volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. **3** shows a block diagram of a timer control with anode **1**, cathode **2**, thermistor temperature sensor **3**, timer control circuit **4** and wire from a direct current power source **5**.

#### EXAMPLE 2

#### Measurement of O<sub>2</sub> Bubbles

Attempts were made to measure the diameter of the  $O_2$ bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be 65 made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured 6

by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

# OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA30** 

60

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 16 of 22

## US RE47,092 E

10

7 TABLE I

17 10 0.0 1				
Gallons	Volts	Amps Max.	Ave	Watts
5	6	0.090	0.060	0.36
32	12	0.180	0.120	1.44
10	12	0.210	0.120	1.44
70	12	0.180	0.180	2.16
2	12	0.180	0.180	2.16
50	12	0.500	0.265	3.48
80	12	0.980	0.410	4.92
2	24	1.200	1.200	28.80
250	12	5.000	2.500	30.00
	5 32 10 70 2 50 80 2	Gallons         Volts           5         6           32         12           10         12           70         12           2         12           50         12           20         12           20         12           20         12           2         2           2         2           2         2	Gallons         Volts         Amps Max.           5         6         0.090           32         12         0.180           10         12         0.210           70         12         0.180           2         12         0.180           50         12         0.500           80         12         0.980           2         24         1.200	Gallons         Volts         Amps Max.         Ave           5         6         0.090         0.060           32         12         0.180         0.120           10         12         0.210         0.120           70         12         0.180         0.180           2         12         0.180         0.180           50         12         0.500         0.265           80         12         0.980         0.410           2         24         1.200         1.200

#### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An O<sub>2</sub> emitter was made in a multilayer sandwich embodiment. (FIG. **5**) An iridium oxide coated platinum anode **1** was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes **2**. Spacing was held at the critical distance by nylon spacers **3**. The embodiment illustrated is held in a cassette **4** which is secured by nylon bolt **5** with a nylon washer **6**. The dimensions selected were:

cathode screen nylon spacer anode grid nylon spacer	0.045 inches thick 0.053 inches thick 0.035 inches thick 0.053 inches thick	
cathode screen	0.045 inches thick,	

for an overall emitter thickness of 0.231 inches thick inches. If a more powerful emitter is desired, it is within the scope <sup>35</sup> of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, the number of layers in the sandwich is limited only by <sup>40</sup> the power requirements acceptable for an application.

#### **EXAMPLE 5**

# Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthe- 50 sis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydro- 55 ponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Fur- 60 thermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the 65 '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air,

8

the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in oneinch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 17 of 22

## US RE47,092 E

9 TABLE II

Week of:	Control, grams tomatoes from eight plants/ cumulative total		Test, grams tomatoes from seven plants/ cumulative total		
July 27	240		400		
August 3	180	420	2910	3310	
August 10	905	1325	1830	5140	
August 17	410	1735	2590	7730	
August 24	3300	5035	2470	10200	
August 31	4150	9175	1580	11780	
September 15	not weighed		3710	15490	

10

any of the emitters shown in FIG. **4** or **5**, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2° C. but the flowing water cooled slightly to 11 or 11.5° C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE III

MODEL	ACTIVE ELECTRODE AREA, SQ.IN.	VOLTAGE	CURRENT, AMPS.	FLOW RATE GAL/MINUTE	DO OF* SAMPLE AT ONE MINUTE
2-Inch "T"	2	28.3	0.72	12	N/A
3-inch "T"	3	28.3	1.75	12	N/A
2-plate Tube	20	28.3	9.1	12	8.4
3-Plate tube	30	28.3	12.8	12	9.6

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The one-minute time point shows the rapid increase in oxygenation.
25

	TABLE II-continued				
Week of:	Control, grams tomatoes from eight plants/ cumulative total		Test, grams tomatoes from seven plants/ cumulative total		
Final Harvest September 24	6435	15620	8895	24385	

The total yield for the eight Control plants was 15620<sup>35</sup> grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. **6** shows the cumulative total as plotted against time. <sup>40</sup> Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and <sup>45</sup> weighed at that point.

#### EXAMPLE 6

#### Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate 55 size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at  $120^{\circ}$  angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is 60 preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will <sup>30</sup> be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the <sup>35</sup> entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

#### EXAMPLE 7

#### Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. **8**, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

# OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

50

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 18 of 22

## US RE47,092 E

11

The invention claimed is:

[1. A method for treating waste water comprising;

providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance 5 from a cathode and a power source all in electrical communication with each other,

placing the emitter within a conduit; and

passing waste water through the conduit.]

[2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breaking the surface tension of the aqueous with oxygen.]

[3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.]

[4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.]

[5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.]

[6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.]

[7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.]

**[8**. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising:

inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.]

**9**. A method for lowering the biologic oxygen demand of polluted water comprising:

passing the polluted water through a vessel containing the emitter of claim 2.]

[10. A supersaturated aqueous product formed with the 45 *electrode*, emitter of claim 2, the supersaturated aqueous product *wherein* having an approximately neutral pH.] gitud

[11. The emitter of claim 2, further comprising a timer control.]

**[12.** The emitter of claim **2**, wherein the anode and 50 cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.]

13. A method for treating water comprising:

providing a flow-through oxygenator comprising an emit-

- ter for electrolytic generation of bubbles of oxygen, the 55 emitter including:
  - a tubular housing having a water inlet, a water outlet, and a longitudinal water flow axis from the inlet to the outlet;
  - at least two electrodes comprising a first electrode and 60 a second electrode, the first and second electrodes being positioned in the tubular housing, the first electrode opposing and separated from the second electrode by a distance of between 0.005 inches to 0.140 inches within the tubular housing; 65
- each electrode of the emitter is positioned so that substantially all points midway between all opposing

12

- electrodes are closer to a surface of the tubular housing than to a center point within the tubular housing and so that at least some water may flow from the water inlet to the water outlet without passing through a space between electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches;
- a power source in electrical communication with the electrodes, the power source configured to deliver a voltage to the electrodes, the voltage being less than or equal to 28.3 volts, the power source being configured to deliver a current to the electrodes, the current being less than or equal to 12.8 amps;
- passing water through the tubular housing while electrical current is applied to the electrodes producing oxygen in said water via electrolysis.

14. The method of claim 13 wherein the tubular housing includes an inward-facing surface that runs parallel to the longitudinal axis;

wherein the first and second electrodes extend in a direction that is parallel to the longitudinal axis.

15. The method of claim 13 wherein the tubular housing includes an inward-facing surface that runs parallel to the 25 longitudinal axis;

- wherein the first and second electrodes extend in a direction parallel to the longitudinal axis; and
- wherein each electrode of the emitter is positioned closer to the inward-facing surface than to the longitudinal axis at the center of the tubular housing.

16. The method of claim 13 wherein at least one of the electrodes is a stainless steel mesh or screen.

17. The method of claim 13 wherein the first and second electrodes are positioned away from a longitudinal center as axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of the first and second electrodes positioned within the tubular housing.

18. The method of claim 17 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing first and second electrodes within the tubular housing.

19. The method of claim 17 wherein the first and second electrodes comprise an outside electrode and an inside electrode,

wherein the first and second electrodes extend in a longitudinal direction parallel to the longitudinal axis and an inward-facing surface of the tubular housing, the outside and inside electrodes being outside and inside electrodes respectively in that the first and second electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal axis at the center of the tubular housing than the outside electrode is.

wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward facing surface of the tubular housing that is substantially less than a cross-sectional area of the unobstructed passageway.

20. The method of claim 13 wherein the first and second electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed 5 passageway parallel to and including the center axis, the passageway running for at least the length of the first and second electrodes positioned within the housing;

# CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 19 of 22

## US RE47,092 E

13

- wherein the first and second electrodes comprise an outside electrode and an inside electrode;
- wherein the first and second electrodes extend in a longitudinal direction parallel to the longitudinal axis and an inward-facing surface of the tubular housing;
- the outside and inside electrodes being outside and inside electrodes respectively in that the first and second electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the 10 inside electrode is closer to the longitudinal axis at the center of the tubular housing than the outside electrode is;
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward 15 facing surface of the tubular housing that is substantially less than a cross-sectional area of the unobstructed passageway; and
- wherein the tubular housing of the emitter is round.
- 21. The method of claim 19 wherein said inward-facing 20 surface is a concave surface.

22. The method of claim 13 further including at least one conductor coupled to one of the first and second electrodes, the at least one conductor exiting a wall of the housing in a radial direction relative to the longitudinal axis of the 25 housing.

23. The method of claim 13 wherein the oxygen produced comprises microbubbles.

24. The method of claim 13 wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps 30 or less per 3 square inches of active electrode.

25. The method of claim 13 wherein the first electrode includes a first anode element and the second electrode includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first 35 anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element.

26. The method of claim 13 wherein the oxygen produced comprises nanobubbles.

 $2^{7}$ . An emitter for electrolytic generation of bubbles of 40 oxygen in water, the emitter comprising:

- a tubular housing defining an oxygenation chamber and having a water inlet, a water outlet, a longitudinal water flow axis from the inlet to the outlet, and an inward-facing surface that runs parallel to the water 45 flow axis and defines at least in part the oxygenation chamber;
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, 50 said outside and inside electrodes extending in a direction that is parallel to the longitudinal axis, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches within the chamber, 55
- wherein the position and size of each electrode within the chamber defines a cross-section of the chamber that has a water flow area within the oxygenation chamber through which water may flow without passing between electrodes of opposite polarity that are separated by a 60 distance of between 0.005 inches to 0.140 inches, wherein the water flow area is greater than an area at the cross-section equal to the total area between electrodes of opposite polarity that are separated by a distance of between 0.005 inches to 0.140 inches, 65
- wherein at least a portion of the outside electrode positioned in the chamber is closer to the inward-facing

14

surface of the oxygenation chamber than to a longitudinal center axis of the oxygenation chamber; and

a power source in electrical communication with the outside and inside electrodes, the power source configured to deliver a voltage to the outside and inside electrodes, the voltage being less than or equal to 28.3 volts, the power source being configured to deliver a current to the outside and inside electrodes, the current being less than or equal to 12.8 amps.

28. The emitter of claim 27 wherein each electrode is positioned closer to the inward-facing surface of the chamber than to the longitudinal center axis of the oxygenation chamber.

29. The emitter of claim 27 wherein the outside and inside electrodes are positioned away from the longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of the outside and inside electrodes positioned within the chamber.

30. The emitter of claim 29 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing inner and outer electrodes within the chamber.

31. The emitter of claim 30 wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward-facing surface of the chamber that is substantially less than a cross-sectional area of said unobstructed passageway.

32. The emitter of claim 27 comprising at least one conductor coupled to one of the outside and inside electrodes, the at least one conductor exiting a wall of the housing in a radial direction relative to the longitudinal center axis of the housing.

33. The emitter of claim 27 wherein the power source is configured to deliver a current to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

34. The emitter of claim 27 wherein the outer electrode includes a first anode element and the inner electrode includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element. 35. A method for treating water comprising:

- providing a flow-through oxygenator comprising an emitter for electrolytic generation of bubbles of oxygen, the emitter including:
  - a tubular housing defining an oxygenation chamber and having a water inlet, and a water outlet;
  - at least two electrodes comprising a first electrode and a second electrode, the first and second electrodes being positioned in the oxygenation chamber, the first electrode opposing and separated from the second electrode by a distance of between 0.005 inches to 0.140 inches, a portion of at least one of the first and second electrodes being in contact with at least one wall of the tubular housing, said wall defining at least in part the oxygenation chamber, wherein each electrode is positioned within the oxygenation chamber so that a cross section of the oxygenation chamber includes a water flow area that allows water to avoid passing between electrodes separated by 0.005 inches to 0.140 inches;
  - a power source in electrical communication with the first and second electrodes, the power source configured to deliver a voltage to the first and second electrodes, the voltage being less than or equal to 28.3 volts, the power source being configured to

## CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 20 of 22

### US RE47,092 E

15

deliver a current to the first and second electrodes, the current being less than or equal to 12.8 amps; passing water through the tubular housing while electrical current is applied to the first and second electrodes to produce oxygen in said water via electrolysis.

36. The method of claim 35 wherein the tubular housing has a longitudinal center axis and an inward-facing surface that runs parallel to the longitudinal center axis; and

wherein each electrode of the emitter is positioned so that substantially all points midway between all opposing 10 electrodes inside the chamber are closer to said inwardly-facing surface than to the longitudinal center axis.

37. The method of claim 35 wherein the chamber has a longitudinal center axis and an inward-facing surface that 15 runs parallel to the longitudinal axis,

wherein the first and second electrodes extend in a direction that is parallel to the longitudinal axis.

38. The method of claim 37 wherein each electrode of the emitter is positioned closer to the inward-facing surface of 20 the chamber than to the longitudinal center axis of the oxygenation chamber.

*39.* The method of claim 35 wherein the at least one electrode in contact with a wall of the tubular housing is in contact with a curved wall of the tubular housing. 25

40. The method of claim 35 wherein the outside and inside electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of the outside 30 and inside electrodes positioned within the chamber.

41. The method of claim 40 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing first and second electrodes within the chamber. 35

42. The method of claim 40 wherein the chamber has an inward-facing surface that runs parallel to the longitudinal axis;

- wherein the first and second electrodes being outside and inside electrodes respectively in that the first and sec-40 ond electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal axis at the center of the tubular housing than the outside electrode 45 is; and
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward facing surface of the tubular housing that is substantially less than a cross-sectional area of the unob- 50 structed passageway.

43. The method of claim 35 wherein the emitter includes at least one conductor coupled to one of the first and second electrodes, the at least one conductor exiting a wall of the housing in a radial direction relative to a longitudinal axis 55 of the housing.

44. The method of claim 35 wherein the oxygen produced comprises microbubbles.

45. The method of claim 35 wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps 60 surface is a concave surface. or less per 3 square inches of active electrode. 55. The method of claim 40

46. The method of claim 35 wherein the first electrode includes a first anode element and the second electrode includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first 65 anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element.

#### 16

47. The method of claim 35 wherein the oxygen produced comprises nanobubbles.

48. A method for treating water comprising:

- providing a flow-through oxygenator comprising an emitter for electrolytic generation of bubbles of oxygen, the emitter including:
  - a tubular housing defining an oxygenation chamber, said housing having an inward-facing surface that defines at least in part the oxygenation chamber, the tubular housing having a water inlet, and a water outlet;
  - at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, said outside and inside electrodes extending in a direction that runs parallel to the inwardfacing surface, the outside and inside electrodes being outside and inside electrodes respectively in that the outside and inside electrodes are positioned relative to each other so that the outside electrode is closer to the inward-facing surface of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal center axis than the outside electrode is, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches within the chamber;
- wherein each electrode of the emitter is positioned closer to the inward-facing surface of the chamber than to a midpoint of the tubular housing and so that at least some water may flow through an unobstructed passageway from the water inlet to the water outlet without passing through a space between electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches;
- passing water through the oxygenation chamber while applying electrical current to the electrodes to produce oxygen in said water via electrolysis.

49. The method of claim 48 wherein the tubular housing defines a longitudinal center axis that lies in the oxygenation chamber and wherein the unobstructed passageway includes the longitudinal center axis.

50. The method of claim 48 wherein at least one of the outside and inside electrodes is in contact with at least one wall of the tubular housing, said wall defining at least in part the oxygenation chamber.

51. The method of claim 50 wherein the at least one electrode in contact with a wall of the tubular housing is in contact with a curved wall of the tubular housing.

52. The method of claim 48 wherein the unobstructed passageway is multiple times wider than the distance separating the opposing inner and outer electrodes within the chamber.

53. The method of claim 52 wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward-facing surface of the chamber that is sub-stantially less than a cross-sectional area of said unob-structed passageway.

54. The method of claim 53 wherein said inward-facing surface is a concave surface.

55. The method of claim 48 wherein the emitter includes at least one conductor coupled to one of the outside and inside electrodes, the at least one conductor exiting a wall of the housing in a radial direction relative to a longitudinal center axis of the housing.

56. The method of claim 48 wherein the oxygen produced comprises microbubbles of oxygen.

## CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 21 of 22

### US RE47,092 E

#### 17

57. The method of claim 48 wherein the electrical current is applied to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

58. The method of claim 48 wherein the outside electrode includes a first anode element and the inside electrode 5 includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element.

59. The method of claim 48 wherein the oxygen produced 10 comprises nanobubbles of oxygen.

60. An emitter for electrolytic generation of bubbles of oxygen in water, the emitter comprising:

- a tubular oxygenation chamber, said chamber having an outer wall that runs parallel to a longitudinal center 15 axis of the chamber, said chamber having a water inlet and a water outlet,
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, the 20 outside and inside electrodes being outside and inside electrodes respectively in that the electrodes are positioned relative to each other so that the outside electrode is closer to the outer wall of the chamber than the inside electrode is and so that the inside electrode is 25 closer to the longitudinal center axis than the outside electrode is, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches;
- the at least two electrodes being positioned away from the 30 center axis and maintaining a longitudinal, unobstructed passageway parallel to and including the center axis that runs for at least the length of the at least two electrodes positioned within the chamber, the unobstructed passageway having a substantially uni-35 form cross-sectional area along that length, the at least two electrodes being positioned so that water may flow from the water inlet to the water outlet without passing through a gistance of between 0.005 inches to 40 0.140 inches;
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the outer wall of the chamber that is substantially less than said cross-sectional area of the unobstructed passageway. 45

61. The emitter of claim 60 wherein at least one of the outside and inside electrodes is in contact with at least one wall of the tubular oxygenation chamber.

62. The emitter of claim 61 wherein the at least one electrode in contact with a wall of the tubular oxygenation 50 chamber is in contact with the outer wall, and wherein the outer wall is a curved wall of the oxygenation chamber.

63. The emitter of claim 60 wherein the unobstructed passageway is multiple times wider than the distance separating the opposing outside and inside electrodes within the 55 chamber.

64. The emitter of claim 60 wherein said outer wall includes an inwardly-facing concave surface.

65. The emitter of claim 60 comprising at least one conductor coupled to one of the outside and inside elec- 60 trodes, the at least one conductor exiting a wall of the chamber in a radial direction relative to the longitudinal center axis of the chamber.

66. The emitter of claim 60 wherein the outside electrode includes a first anode element and the inside electrode 65 includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first

#### 18

- anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element. 67. A method for treating water comprising:
- providing a flow-through oxygenator comprising an emitter for electrolytic generation of bubbles of oxygen, the emitter including:
  - a tubular housing defining an oxygenation chamber and having a water inlet, a water outlet, a longitudinal water flow axis from the inlet to the outlet, and an inward-facing surface that runs parallel to the water flow axis and defines at least in part the oxygenation chamber;
  - at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, said outside and inside electrodes extending in a direction that is parallel to the longitudinal axis, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches within the chamber,
  - wherein the position and size of each electrode within the chamber defines a cross-section of the chamber that has a water flow area within the oxygenation chamber through which water may flow without passing between electrodes of opposite polarity that are separated by a distance of between 0.005 inches to 0.140 inches, wherein the water flow area is greater than an area at the cross-section equal to the total area between electrodes of opposite polarity that are separated by a distance of between 0.005 inches to 0.140 inches; and
  - a power source in electrical communication with the outside and inside electrodes, the power source configured to deliver a voltage to the outside and inside electrodes, the voltage being less than or equal to 28.3 volts, the power source being configured to deliver a current to the outside and inside electrodes, the current being less than or equal to 12.8 amps;
- passing water through the oxygenation chamber while electrical current is applied to the outside and inside electrodes within the chamber to produce oxygen in said water via electrolysis.

68. The method of claim 67 wherein each electrode of the emitter is positioned closer to the inward-facing surface of the chamber than to a longitudinal center axis of the oxygenation chamber.

69. The method of claim 67 wherein the outside and inside electrodes of the emitter is positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the longitudinal center axis, the passageway running longitudinally for at least the length of the outside and inside electrodes within the chamber.

70. The method of claim 69 wherein the unobstructed passageway includes the longitudinal center axis and is multiple times wider than the distance separating the opposing inner and outer electrodes within the chamber.

71. The method of claim 70 wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward-facing surface of the chamber that is substantially less than a cross-sectional area of said unobstructed passageway.

72. The method of claim 67 wherein the emitter includes at least one conductor coupled to one of the outside and

## CASE 0:20-cv-00358-ECT-HB Document 9-2 Filed 05/08/20 Page 22 of 22

### US RE47,092 E

#### 19

inside electrodes, the at least one conductor exiting a wall of the housing in a radial direction relative to a longitudinal center axis of the housing.

73. The method of claim 67 wherein the oxygen produced comprises nanobubbles.

74. The method of claim 67 wherein the power source delivers a current to the outside and inside electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

75. The method of claim 67 wherein the outside electrode 10 includes a first anode element and the inside electrode includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element. 15

76. The method of claim 75 wherein the oxygen produced comprises nanobubbles.

77. A method for treating water comprising:

- providing a flow-through oxygenator comprising an emitter for electrolytic generation of bubbles of oxygen, the 20 emitter including:
- a tubular oxygenation chamber, said chamber having an outer wall that runs parallel to a longitudinal center axis of the chamber, said chamber having a water inlet and a water outlet;
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, the outside and inside electrodes being outside and inside electrodes respectively in that the 30 outside and inside electrodes are positioned relative to each other so that the outside electrode is closer to the outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal center axis than the outside 35 electrode is, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches;
- the at least two electrodes of the emitter being positioned away from the longitudinal center axis and 40 maintaining a longitudinal, unobstructed passageway parallel to and including the longitudinal center axis that runs for at least the length of the at least two electrodes positioned within the chamber, the

20

- unobstructed passageway having a substantially uniform cross-sectional area along that length, the at least two electrodes of the emitter being positioned so that water may flow from the water inlet to the water outlet without passing through a space between electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches;
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the outer wall of the chamber that is substantially less than said cross-sectional area of the unobstructed passageway; and
- passing water through the oxygenation chamber while applying electrical current to the outside and inside electrodes to produce oxygen in said water via electrolysis.

78. The method of claim 77 wherein at least one of the outside and inside electrodes is in contact with at least one wall of the tubular oxygenation chamber.

79. The method of claim 78 wherein the at least one electrode in contact with a wall of the tubular oxygenation chamber is in contact with the outer wall, and wherein the outer wall is a curved wall of the oxygenation chamber.

80. The method of claim 77 wherein the unobstructed passageway is multiple times wider than the distance separating the opposing outside and inside electrodes within the chamber.

81. The method of claim 77 wherein said outer wall includes an inwardly-facing concave surface.

82. The method of claim 77 wherein the emitter includes at least one conductor coupled to one of the outside and inside electrodes, the at least one conductor exiting a wall of the chamber in a radial direction relative to the longitudinal center axis of the chamber.

83. The method of claim 77 wherein the outside electrode includes a first anode element and the inside electrode includes a first cathode element, and wherein the emitter includes a second anode element non-parallel to the first anode element, and wherein the emitter includes a second cathode element non-parallel to the first cathode element.

84. The method of claim 83 wherein the oxygen produced comprises nanobubbles of oxygen.

\* \* \* \* \*

CASE 0:20-cv-00358-ECT-HB Docu



## (19) United States

## (12) **Reissued Patent** (10) Patent Number: Senkiw (45) Date of Reissued

# (10) Patent Number:USRE47,665 E(45) Date of Reissued Patent:\*Oct. 29, 2019

### (54) FLOW-THROUGH OXYGENATOR

- (71) Applicant: Oxygenator Water Technologies, Inc., St. Louis Park, MN (US)
- (72) Inventor: James Andrew Senkiw, St. Louis Park, MN (US)
- (\*) Notice: This patent is subject to a terminal disclaimer.
- (21) Appl. No.: 14/601,340
- (22) Filed: Jan. 21, 2015

**Related U.S. Patent Documents** 

Reiss	ue of:	
(64)	Patent No.:	7,670,495
	Issued:	Mar. 2, 2010
	Appl. No.:	12/023,431
	Filed:	Jan. 31, 2008

U.S. Applications:

- (60) Continuation of application No. 13/247,241, filed on Sep. 28, 2011, now Pat. No. Re. 45,415, which is an (Continued)
- (51) Int. Cl. *A01G 31/02* (2006.01) *A01K 63/04* (2006.01)

(Continued) Field of Classification Search

(58) Field of Classification Search CPC ....... A01G 31/02; A01G 31/00; C02F 1/727; C02F 1/46109; C02F 3/26; C02F 7/00; (Continued)

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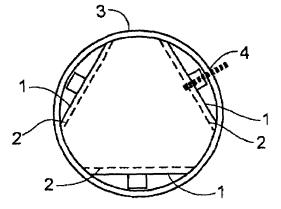
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Primary Examiner — Jerry D Johnson (74) Attorney, Agent, or Firm — Carlson Caspers Vandenburgh & Lindquist, PA

#### (57) ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

#### 49 Claims, 8 Drawing Sheets



## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 3 of 21

## US RE47,665 E

Page 2

### **Related U.S. Application Data**

application for the reissue of Pat. No. 7,670,495, which is a division of application No. 10/732,326, filed on Dec. 10, 2003, now Pat. No. 7,396,441, which is a continuation-in-part of application No. 10/372,017, filed on Feb. 21, 2003, now Pat. No. 6,689,262.

- (60) Provisional application No. 60/358,534, filed on Feb. 22, 2002.
- (51)

Int. Cl.	
C02F 1/461	(2006.01)
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C02F 3/26	(2006.01)
A01G 31/00	(2018.01)
C02F 1/467	(2006.01)
C02F 1/68	(2006.01)
C02F 7/00	(2006.01)

(52) U.S. Cl.

CPC ...... C02F 1/46109 (2013.01); C02F 1/727 (2013.01); C02F 3/26 (2013.01); C02F 1/4672 (2013.01); C02F 1/68 (2013.01); C02F 7/00 (2013.01); C02F 2001/46133 (2013.01); C02F 2001/46138 (2013.01); C02F 2001/46157 (2013.01); C02F 2201/4612 (2013.01); C02F 2201/4615 (2013.01); C02F 2209/02 (2013.01); Y02E 60/366 (2013.01); Y02P 60/216 (2015.11); Y02W 10/15 (2015.05)

(58) Field of Classification Search

CPC ...... C02F 2001/46138; C02F 2209/02; C02F 2001/46133; C02F 2001/46157; C02F 1/4672; C02F 1/68; C02F 2201/4612; C02F 2201/4615; A01K 63/042; Y02P 60/216; Y02W 10/15; Y02E 60/366 See application file for complete search history.

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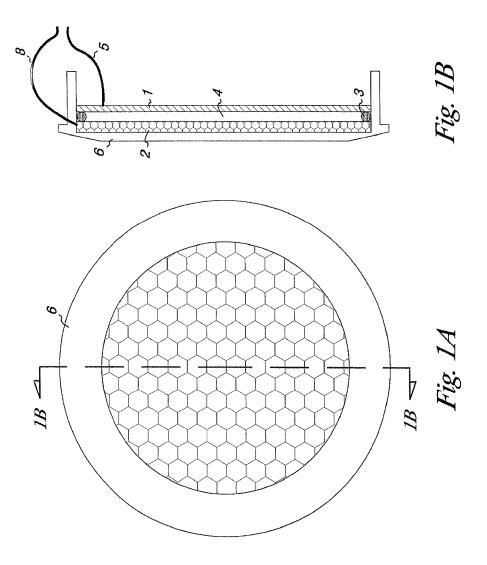
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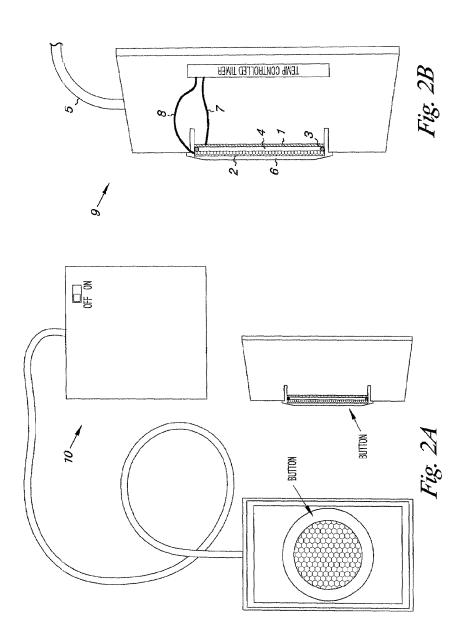
CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 5 of 21

U.S. Patent Oct. 29, 2019 Sheet 1 of 8 US RE47,665 E



CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 6 of 21

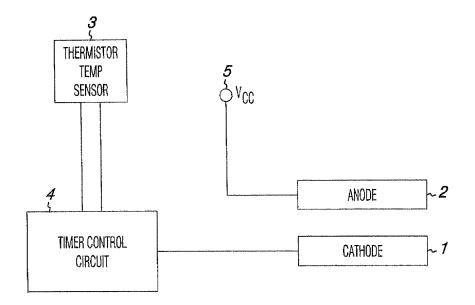
U.S. Patent Oct. 29, 2019 Sheet 2 of 8 US RE47,665 E



## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 45 of 1320

CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 7 of 21

U.S. Patent Oct. 29, 2019 Sheet 3 of 8 US RE47,665 E

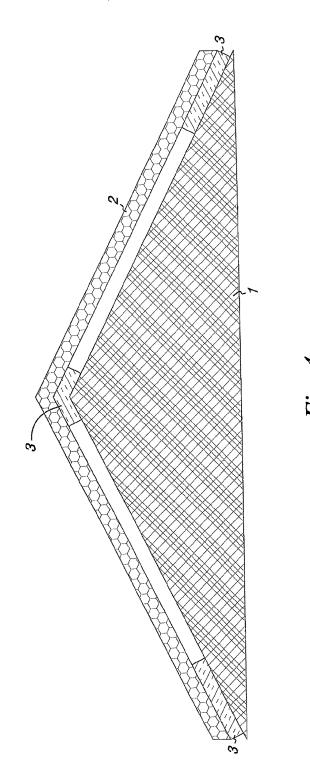


*Fig. 3* 

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CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 8 of 21

U.S. Patent Oct. 29, 2019 Sheet 4 of 8 US RE47,665 E



## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 47 of 1320

CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 9 of 21

U.S. Patent Oct. 29, 2019 Sheet 5 of 8 US RE47,665 E

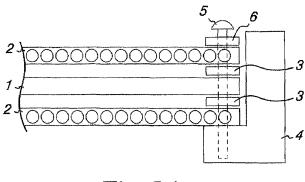
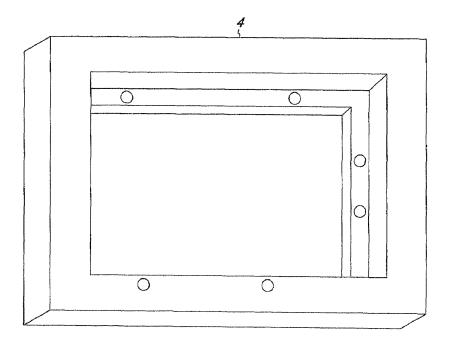


Fig. 5A



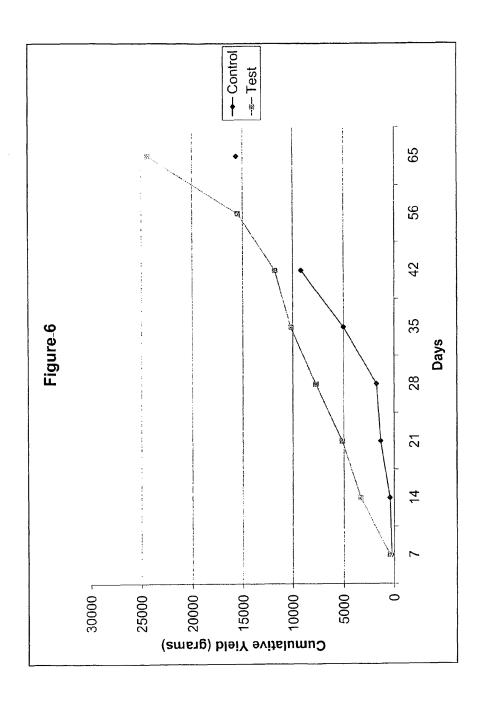
*Fig.* 5*B* 

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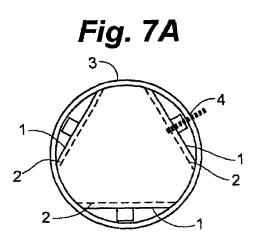
CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 10 of 21

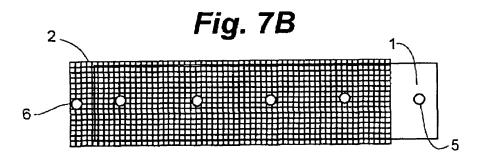
U.S. Patent Oct. 29, 2019 Sheet 6 of 8 US RE47,665 E



CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 11 of 21

U.S. Patent Oct. 29, 2019 Sheet 7 of 8 US RE47,665 E



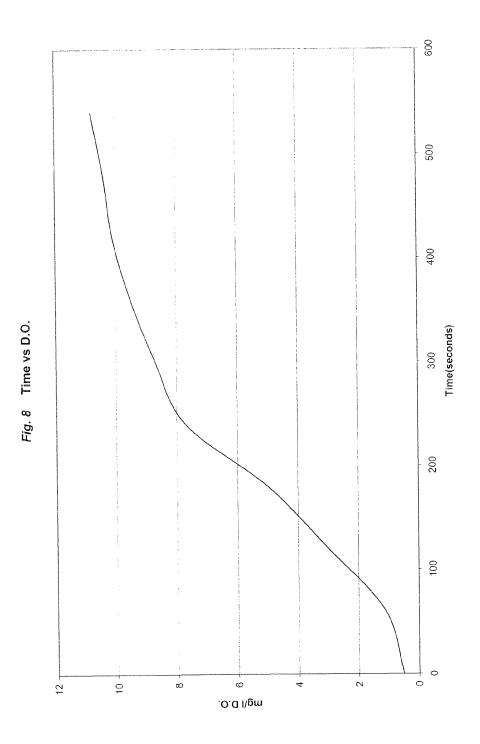


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CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 12 of 21

U.S. Patent Oct. 29, 2019 Sheet 8 of 8 US RE47,665 E





**JA48** 

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 13 of 21

## US RE47,665 E

#### 1 FLOW-THROUGH OXYGENATOR

Matter enclosed in heavy brackets [] appears in the original patent but forms no part of this reissue specification; matter printed in italics indicates the additions made by reissue; a claim printed with strikethrough indicates that the claim was canceled, disclaimed, or held invalid by a prior post-patent action or proceeding.

#### RELATED APPLICATIONS

More than one reissue application has been filed for the reissue of U.S. Pat. No. 7,670,495. This application a continuation reissue application of application Ser. No. 15 13/247,241, filed Sep. 28, 2011, now U.S. Pat. No. RE45, 415, which is a reissue of U.S. Pat. No. 7,670,495. U.S. Pat. No. 6,760,495 is a division of application Ser. No. 10/732, 326 filed Dec. 10, 2003, which in turn is a continuation-inpart of application Ser. No. 10/372,017, filed Feb. 21, 2003, <sup>20</sup> now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of <sup>30</sup> superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for 40 applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an 45 aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of 50 the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct 55 chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms. 60

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size 65 of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. 2

Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$
NET REACTION:	$6H_2O \rightarrow 4OH^- + 4H^+ + 2H_2 + O_2$

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 14 of 21

## US RE47,665 E

3

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the 10 medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a {fraction  $(\frac{1}{16})$ } inch 20 microbubbles and nanobubbles. grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. 25 50 microns. The preferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those 30 models with low voltage requirements are especially suited to oxygenating water in which animals are to be held. Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be con- 35 nected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alterna- 40 tively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow 45 rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated 50 water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the  $O_2$  emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the  $O_2$ emitter.

FIG. 4 shows a funnel or pyramid variation of the  $O_2$ emitter.

FIG. 5 shows a multilayer sandwich O<sub>2</sub> emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

4

FIG. 7 shows an oxygenation chamber suitable for flowthrough applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source

FIG. 8 is a graph showing the oxygenation of waste water.

#### DETAILED DESCRIPTION OF THE INVENTION

Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms

'O2 emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O2. In the special dimensions of the invention, as explained in more detail in the following examples, O2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are 55 discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a 60 small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while 65 hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode.

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 15 of 21

## US RE47,665 E

5 Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided 1 sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction  $(\frac{1}{16})$ } inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic 1 and cathodic water and connected to a power source through a connection point 5. FIG. 2 shows a plan view of the assembled device. The O2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. 20 The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher 25 voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, 30 Teflon®, polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a noncompressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure 33 of 90 and was found to hold its shape well.

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This <sup>40</sup> size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off <sup>50</sup> time is shorter. Thus the device is self-controlled to use power most economically. FIG. **3** shows a block diagram of a timer control with anode **1**, cathode **2**, thermistor temperature sensor **3**, timer control circuit **4** and wire from a direct current power source **5**.

#### EXAMPLE 2

#### Measurement of O<sub>2</sub> Bubbles

Attempts were made to measure the diameter of the  $O_2$  bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured 65 by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital,

6

high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that 55 a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to 60 various uses.

TABLE	Ι	
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	Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts
5	Bait keeper	5	6	0.090	0.060	0.36
	Livewell	32	12	0.180	0.120	1.44

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 16 of 21

## US RE47,665 E

7 TABLE I-continued

Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5.000	2.500	30.00

#### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An O<sub>2</sub> emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2.  $_{20}$ Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

#### EXAMPLE 5

#### Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the 50 roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxy-gen deficit in the root system. U.S. Pat. No. 5,887,383 55 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a 60 report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, 65 the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

8

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the appli-<sup>25</sup> cation of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in oneinch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of  $_{\rm 40}~$  Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off. about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

> **OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

45

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 17 of 21

## US RE47,665 E

TABLE II

9

10 TABLE III

Week of:	Control, g tomatoes fror plants/cumulat	n eight	tomatoes	grams from seven ulative total	
July 27	240		400		-
August 3	180	420	2910	3310	
August 10	905	1325	1830	5140	
August 17	410	1735	2590	7730	1
August 24	3300	5035	2470	10200	,
August 31	4150	9175	1580	11780	
September 15	not weighed		3710	15490	
Final Harvest	6435	15620	8895	24385	
September 24					1

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams 20 or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

#### EXAMPLE 6

#### Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120° angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can  $_{45}$ be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power 50 source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other 55 embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage  $_{60}$ and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2° C. but the flowing water cooled slightly to 11 or 11.5° C. Without undue experimentation, anyone may easily select the embodiment that best 65 separated at a critical distance from a cathode, a nonconsuits desired characteristics from Table III or designed with the teachings of Table III.

MODEL	ACTIVE ELECTRODE AREA, SQ. IN.	VOLT- AGE	CUR- RENT, AMPS.	FLOW RATE GAL/ MINUTE	DO OF* SAMPLE AT ONE MINUTE
2-Inch "T"	2	28.3	0.72	12	N/A
3-inch "T"	3	28.3	1.75	12	N/A
2-plate Tube	20	28.3	9.1	12	8.4
3-Plate tube	30	28.3	12.8	12	9.6

As the apparatus runs longer, the flowing water becomes milky, indicating supersatura-ion. The one-minute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants 30 and to adjust their protocols accordingly.

It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water 35 only once a day.

#### EXAMPLE 7

#### Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims

The invention claimed is:

- [1. A method for treating waste water comprising;
- providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other,

placing the emitter within a conduit; and

passing waste water through the conduit.]

[2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode ductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness

## **OWT Ex. 2118 Tennant Company v. OWT**

IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 18 of 21

### US RE47,665 E

between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breaking the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.]

[3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic 10 oxide.

[4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.]

[5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic 15 oxide.]

[6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.]

[7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of 20 electrodes is a stainless steel mesh or screen. cathodes.

[8. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising:

inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait 25 bucket or a live well.

9. A method for lowering the biologic oxygen demand of polluted water comprising:

passing the polluted water through a vessel containing the emitter of claim 2.

[10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.]

[11. The emitter of claim 2, further comprising a timer control.

[12. The emitter of claim 2, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.]

13. An emitter for electrolytic generation of bubbles of oxygen in water, the emitter comprising:

- a tubular housing having a water inlet, a water outlet, and a longitudinal water flow axis from the inlet to the outlet
- at least two electrodes comprising a first electrode and a second electrode, the first and second electrodes being 45 positioned in the tubular housing, the first electrode opposing and separated from the second electrode by a distance of between 0.005 inches to 0.140 inches within the tubular housing;
- each electrode of the emitter is positioned so that all 50 points midway between all opposing electrodes are closer to a surface of the tubular housing than to a center point within the tubular housing and so that at least some water may flow from the water inlet to the water outlet without passing through a space between 55 electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches;
- power source in electrical communication with the electrodes, the power source configured to deliver a voltage to the electrodes, the voltage being less than or 60 equal to 28.3 volts, the power source being configured to deliver a current to the electrodes, the current being less than or equal to 12.8 amps;
- the power source being operable to deliver electrical current to the electrodes while water flows through the 65 tubular housing and is in contact with the electrodes to produce oxvgen in said water via electrolysis.

#### 12

14. The emitter of claim 13 wherein the tubular housing includes an inward-facing surface that runs parallel to the longitudinal axis:

- wherein the electrodes extend in a direction that is parallel to the longitudinal axis; and
- wherein at least one of the first and second electrodes is positioned in the tubular housing closer to the inwardfacing surface than said distance separating the electrodes.

15. The emitter of claim 13 wherein the tubular housing includes an inward-facing surface that runs parallel to the longitudinal axis:

- wherein said electrodes extend in a direction parallel to the longitudinal axis; and
- wherein each electrode of the emitter is positioned closer to the inward-facing surface than to the longitudinal axis at the center of the tubular housing.

16. The emitter of claim 13 wherein at least one of the

17. The emitter of claim 13 wherein the electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of one of the electrodes positioned within the tubular housing.

18. The emitter of claim 17 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing first and second electrodes within the tubular housing.

19. The emitter of claim 17 wherein the first and second electrodes comprise an outside electrode and an inside electrode, wherein the first and second electrodes extend in a longitudinal direction parallel to the longitudinal axis and an inward-facing surface of the tubular housing, the outside and inside electrodes being outside and inside electrodes respectively in that the electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal axis at the center of the tubular housing than the outside electrode is, wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward facing surface of the tubular housing that is less than a cross-sectional area of the unobstructed passageway.

20. The emitter of claim 13 wherein the electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to and including the center axis, the passageway running for at least the length of one of the electrodes positioned within the housing;

- wherein the first and second electrodes comprise an outside electrode and an inside electrode;
- wherein the first and second electrodes extend in a longitudinal direction parallel to the longitudinal axis and an inward-facing surface of the tubular housing;
- the outside and inside electrodes being outside and inside electrodes respectively in that the electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal axis at the center of the tubular housing than the outside electrode is;
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 19 of 21

## US RE47,665 E

13

facing surface of the tubular housing that is less than a cross-sectional area of the unobstructed passageway; and

wherein the tubular housing of the emitter is round.

21. The emitter of claim 19 wherein said inward-facing 5 surface is a concave surface.

22. The emitter of claim 13 further including first and second conductors coupled to the first and second electrodes respectively, the first conductor exiting a wall of the housing in a radial direction relative to the longitudinal axis of the housing, the second conductor exiting a wall of the housing in a radial direction relative to the longitudinal axis of the housing.

23. The emitter of claim 13 wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

24. The emitter of claim 13 wherein the at least two electrodes includes a first anode electrode portion that is nonparallel to a second anode electrode portion, the first 20 and second anode electrode portions each being parallel to respective opposing cathode electrode portions.

25. An emitter for electrolytic generation of bubbles of oxygen in water, the emitter comprising:

- a tubular housing defining an oxygenation chamber and 25 having a water inlet, a water outlet, a longitudinal water flow axis from the inlet to the outlet, and an inward-facing surface that runs parallel to the water flow axis and defines at least in part the oxygenation chamber:
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber and extending in a direction that is parallel to the longitudinal axis, the outside electrode opposing and 35 separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches within the chamber, wherein the position and size of each electrode within the chamber defines a cross-section of the chamber that has a water flow area within the oxygenation 40 chamber through which water may flow without passing between electrodes of opposite polarity that are separated by a distance of between 0.005 inches to 0.140 inches, wherein the water flow area is greater than an area at the cross-section equal to the total area 45 between electrodes of opposite polarity that are separated by a distance of between 0.005 inches to 0.140 inches, wherein at least a portion of the outside electrode positioned in the chamber is closer to the inwardfacing surface of the oxygenation chamber than said 50 distance separating the inside electrode from the outside electrode: and
- a power source in electrical communication with the electrodes, the power source configured to deliver a voltage to the electrodes, the voltage being less than or 55 equal to 28.3 volts, the power source being configured to deliver a current to the electrodes, the current being less than or equal to 12.8 amps;
- the power source being operable to deliver electrical current to the electrodes while water flows through the 60 chamber of the tubular housing and is in contact with the electrodes to produce oxygen in said water via electrolysis.

26. The emitter of claim 25 wherein each electrode of the emitter is positioned closer to the inward-facing surface of 65 that runs parallel to the longitudinal center axis; and the chamber than to a longitudinal center axis of the oxygenation chamber.

#### 14

27. The emitter of claim 25 wherein the electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of one of the electrodes positioned within the chamber.

28. The emitter of claim 27 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing inner and outer electrodes within the chamber.

29. The emitter of claim 28 wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward-facing surface of the chamber that is less than a cross-sectional area of said unobstructed passagewav.

30. The emitter of claim 25 further including first and second conductors coupled to the outside and inside electrodes respectively, the first conductor exiting a wall of the housing in a radial direction relative to a longitudinal center axis of the housing, the second conductor exiting a wall of the housing in a radial direction relative to a longitudinal center axis of the housing.

31. The emitter of claim 25 wherein the oxygen produced comprises nanobubbles.

32. The emitter of claim 25 wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

33. The emitter of claim 25 wherein the at least two electrodes includes a first anode electrode portion that is nonparallel to a second anode electrode portion, the first and second anode electrode portions each being parallel to respective opposing cathode electrode portions

34. An emitter for electrolytic generation of bubbles of oxygen in water, the emitter comprising:

- a tubular housing defining an oxygenation chamber and having a water inlet, and a water outlet;
- at least two electrodes comprising a first electrode and a second electrode, the first and second electrodes being positioned in the oxygenation chamber, the first electrode opposing and separated from the second electrode by a distance of between 0.005 inches to 0.140 inches, a portion of at least one of the first and second electrodes being in contact with at least one wall of the tubular housing, said wall defining at least in part the oxygenation chamber, said portion being a portion that opposes the other of the first and second electrodes, wherein each electrode is positioned within the oxygenation chamber so that a cross section of the oxygenation chamber includes a water flow area that allows water to avoid passing between electrodes separated by 0.005 inches to 0.140 inches;
- a power source in electrical communication with the electrodes, the power source configured to deliver a voltage to the electrodes, the voltage being less than or equal to 28.3 volts, the power source being configured to deliver a current to the electrodes, the current being less than or equal to 12.8 amps;
- the power source being operable to deliver electrical current to the electrodes while water flows through the tubular housing and is in contact with the electrodes to roduce oxygen in said water via electrolysis.

35. The emitter of claim 34 wherein the tubular housing has a longitudinal center axis and an inward-facing surface wherein each electrode of the emitter is positioned so that

all points midway between all opposing electrodes

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 20 of 21

### US RE47,665 E

#### 15

inside the chamber are closer to said inwardly-facing surface than to the longitudinal center axis.

36. The emitter of claim 34 wherein the chamber has a longitudinal center axis and an inward-facing surface that runs parallel to the longitudinal axis, wherein the electrodes 5 extend in a direction that is parallel to the longitudinal axis, and wherein at least one of the first and second electrodes is positioned in the chamber closer to the inward-facing surface than said distance separating the electrodes.

37. The emitter of claim 36 wherein each electrode of the 10 emitter is positioned closer to the inward-facing surface of the chamber than to the longitudinal center axis of the oxygenation chamber.

 $\overline{38}$ . The emitter of claim 34 wherein the electrode in contact with a wall of the tubular housing is in contact with 15 a curved wall of the tubular housing.

39. The emitter of claim 34 wherein the electrodes are positioned away from a longitudinal center axis of the tubular housing and maintain an unobstructed passageway parallel to the center axis, the passageway running longitudinally for at least the length of one of the electrodes positioned within the chamber.

40. The emitter of claim 39 wherein the unobstructed passageway includes the center axis and is multiple times wider than the distance separating the opposing first and 25 second electrodes within the chamber.

41. The emitter of claim 39 wherein the chamber has an inward-facing surface that runs parallel to the longitudinal axis;

- wherein the first and second electrodes being outside and 30 inside electrodes respectively in that the electrodes are positioned relative to each other so that the outside electrode is closer to an outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal axis at the center of the 35 tubular housing than the outside electrode is; and
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward facing surface of the tubular housing that is less than a cross-sectional area of the unobstructed passageway. 40

42. The emitter of claim 34 further including first and second conductors coupled to the first and second electrodes respectively, the first conductor exiting a wall of the housing in a radial direction relative to a longitudinal axis of the housing, the second conductor exiting a wall of the housing 45 in a radial direction relative to the longitudinal axis of the housing.

43. The emitter of claim 34 wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode. 50

44. The emitter of claim 34 wherein the at least two electrodes includes a first anode electrode portion that is nonparallel to a second anode electrode portion, the first and second anode electrode portions each being parallel to respective opposing cathode electrode portions.

45. An emitter for electrolytic generation of bubbles of oxygen in an aqueous medium comprising:

- a tubular housing defining an oxygenation chamber, and having an inward-facing surface that defines at least in part the oxygenation chamber, a water inlet, and a 60 water outlet;
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber and extending in a direction that runs parallel to the 65 inward-facing surface, the outside and inside electrodes being outside and inside electrodes respectively

#### 16

in that the electrodes are positioned relative to each other so that the outside electrode is closer to the inward-facing surface of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal center axis than the outside electrode is, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches within the chamber;

wherein each electrode of the emitter is positioned closer to the inward-facing surface of the chamber than to a midpoint of the tubular housing and so that at least some water may flow through an unobstructed passageway from the water inlet to the water outlet without passing through a space between electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches.

46. The emitter of claim 45 wherein at least one of the inside and outside electrodes is positioned in the chamber closer to the inward-facing surface than said distance separating the electrodes, and wherein the tubular housing defines a longitudinal center axis that lies in the oxygenation chamber and wherein the unobstructed passageway includes the longitudinal center.

47. The emitter of claim 45 wherein at least one of the outside and inside electrodes is in contact with at least one wall of the tubular housing, said wall defining at least in part the oxygenation chamber.

48. The emitter of claim 47 wherein the electrode in contact with a wall of the tubular housing is in contact with a curved wall of the tubular housing.

49. The emitter of claim 45 wherein the unobstructed passageway is multiple times wider than the distance separating the opposing inner and outer electrodes within the chamber.

50. The emitter of claim 49 wherein the outside electrode defines a cross-sectional area between the outside electrode and the inward-facing surface of the chamber that is less than a cross-sectional area of said unobstructed passage-way.

51. The emitter of claim 50 wherein said inward-facing surface is a concave surface.

52. The emitter of claim 45 further including first and second conductors coupled to the outside and inside electrodes respectively, the first conductor exiting a wall of the housing in a radial direction relative to a longitudinal center axis of the housing, the second conductor exiting a wall of the housing in a radial direction relative to the longitudinal center axis of the housing.

53. The emitter of claim 45 coupled to a power source wherein the power source delivers a current to the electrodes at a ratio of 1.75 amps or less per 3 square inches of active electrode.

54. The emitter of claim 45 wherein the at least two electrodes includes a first anode electrode portion that is 55 nonparallel to a second anode electrode portion, the first and second anode electrode portions being parallel to respective opposing cathode electrode portions.

55. An emitter for electrolytic generation of bubbles of oxygen in an aqueous medium comprising:

- a tubular housing defining an oxygenation chamber, said housing having an outer wall that runs parallel to a longitudinal center axis of the housing, said housing having a water inlet and a water outlet,
- at least two electrodes comprising an outside electrode and an inside electrode, the outside and inside electrodes being positioned in the oxygenation chamber, the outside and inside electrodes being outside and inside

## CASE 0:20-cv-00358-ECT-HB Document 9-3 Filed 05/08/20 Page 21 of 21

### US RE47,665 E

17

electrodes respectively in that the electrodes are positioned relative to each other so that the outside electrode is closer to the outer wall of the chamber than the inside electrode is and so that the inside electrode is closer to the longitudinal center axis than the outside electrode is, the outside electrode opposing and separated from the inside electrode by a distance of between 0.005 inches to 0.140 inches;

- the electrodes being positioned away from the center axis and maintaining a longitudinal, unobstructed passageway parallel to and including the center axis that runs for at least the length of one of the electrodes positioned within the chamber, the unobstructed passageway having a uniform cross-sectional area along that length, the electrodes being positioned so that water may flow from the water inlet to the water outlet 15 without passing through a space between electrodes of opposite polarity separated by a distance of between 0.005 inches to 0.140 inches;
- wherein the outside electrode defines a cross-sectional area between the outside electrode and the outer wall 20 of the chamber that is less than said cross-sectional area of the unobstructed passageway.
  56. The emitter of claim 55 wherein at least one of the

56. The emitter of claim 55 wherein at least one of the outside and inside electrodes is in contact with at least one wall of the tubular housing, said wall defining at least in part the oxygenation chamber.

18

57. The emitter of claim 56 wherein the electrode in contact with a wall of the tubular housing is in contact with the outer wall which is a curved wall of the tubular housing.

58. The emitter of claim 55 wherein the unobstructed passageway is multiple times wider than the distance separating the opposing outside and inside electrodes within the chamber.

59. The emitter of claim 55 wherein said outer wall 10 includes an inwardly-facing concave surface.

60. The emitter of claim 55 further including first and second conductors coupled to the outside and inside electrodes respectively, the first conductor exiting a wall of the housing in a radial direction relative to the longitudinal center axis of the housing, the second conductor exiting a wall of the housing in a radial direction relative to the longitudinal center axis of the housing.

61. The emitter of claim 55 wherein the at least two electrodes includes a first anode electrode portion that is nonparallel to a second anode electrode portion, the first and second anode electrode portions each being parallel to respective opposing cathode electrode portions.

\* \* \* \* \*

## (12) United States Patent Senkiw

### (54) MICROBUBBLES OF OXYGEN

- (75) Inventor: James Andrew Senkiw, Minneapolis, MN (US)
- (73) Assignee: Aqua Innovation, Inc., Bloomington, MN (US)
- (\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.
- (21) Appl. No.: 10/372,017
- (22) Filed: Feb. 21, 2003

#### (65) Prior Publication Data

US 2003/0164306 A1 Sep. 4, 2003

## Related U.S. Application Data

- (60) Provisional application No. 60/358,534, filed on Feb. 22, 2002.
- (51) Int. Cl.<sup>7</sup> ..... C25B 9/00

### (56) References Cited

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# (10) Patent No.: US 6,689,262 B2 (45) Date of Patent: Feb. 10, 2004

5,049,252	Α	*	9/1991	Wright
5,534,143 6,315,886	A B1	* *	7/1996 11/2001	Goodman         209/164           Portier et al.         210/151           Zappi et al.         205/701           Ganan-Calvo         261/77
				Greenberg et al 210/748

FOREIGN PATENT DOCUMENTS

WO 95/21795 \* 8/1995

\* cited by examiner

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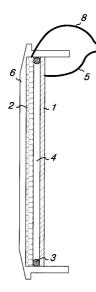
Primary Examiner-Bruce F. Bell

(74) Attorney, Agent, or Firm-Kathleen R. Terry

#### (57) ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium. Models suitable for different uses are disclosed.

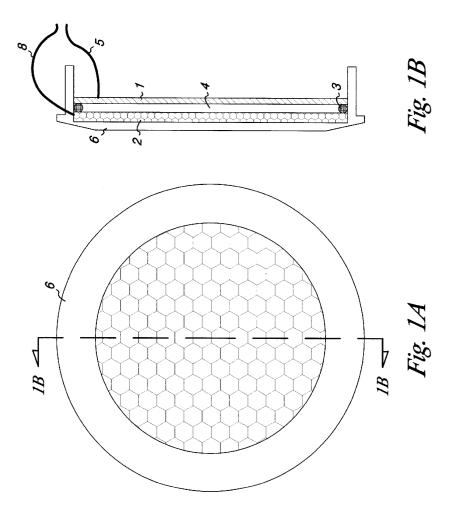
#### 14 Claims, 5 Drawing Sheets



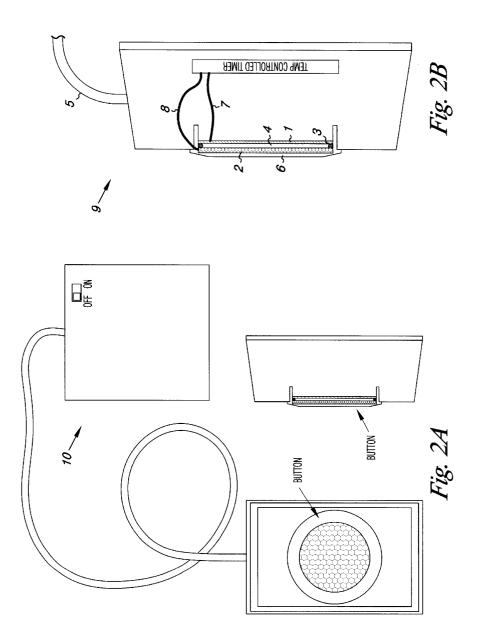


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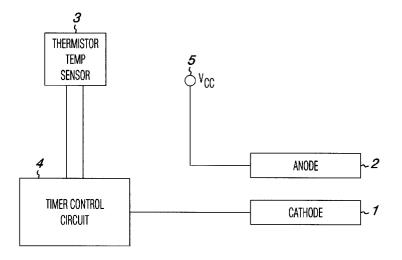
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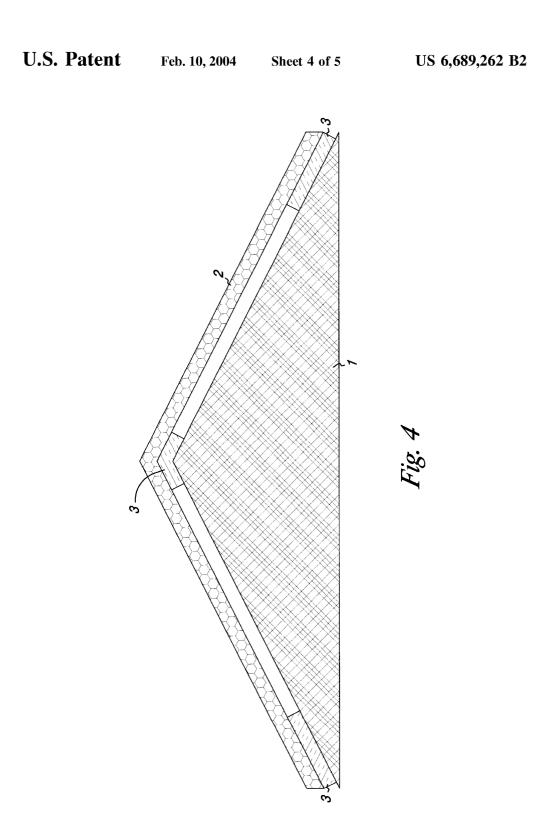




U.S. Patent Feb. 10, 2004 Sheet 3 of 5 US 6,689,262 B2



*Fig. 3* 





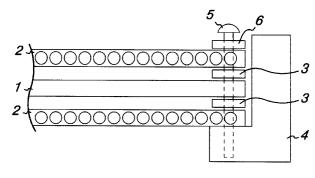


Fig. 5A

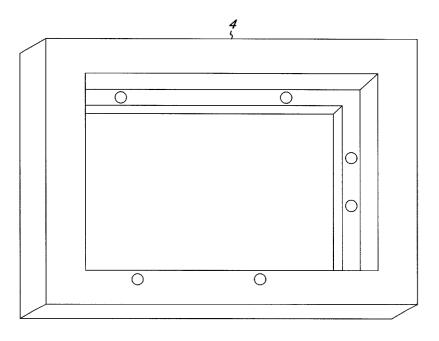


Fig. 5B

## 1 MICROBUBBLES OF OXYGEN

#### RELATED APPLICATIONS

This application claim the priority of U.S. Provisional Patent Application No. 60/358,534, filed Feb. 22, 2002.

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content 10 metallic oxide of at least one metal selected from of aqueous media.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to 15 achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For 20 example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the 25 respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of 30 these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into 40 the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3  $^{45}$ millimeters in diameter. U.S. Pat. No. 6,394,429 discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the 50water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE: AT THE ANODE:	$\begin{array}{l} 4 \ \mathrm{H_2O} + 4 \ \mathrm{e}^- \rightarrow 4 \ \mathrm{OH}^- + 2 \ \mathrm{H_2} \\ 2 \ \mathrm{H_2O} \rightarrow \mathrm{O_2} + 4 \ \mathrm{H}^+ + 4 \ \mathrm{e}^- \end{array}$
NET REACTION:	$6 \text{ H}_2\text{O} \rightarrow 4 \text{ OH}^- + 4 \text{ H}^+ + 2 \text{ H}_2 + \text{O}_2$

286 kilojoules of energy is required to generate one mole of oxygen.

2

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from allovs of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited 65 to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

## **OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

55

3

DESCRIPTION OF THE DRAWINGS

FIG. 1 is the  $O_2$  emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the  $\mathrm{O}_2_{\phantom{2}5}$ 

FIG. 4 shows a funnel or pyramid variation of the  $\rm O_2$  emitter.

FIG. 5 shows a multilayer sandwich O<sub>2</sub> emitter.

#### DETAILED DESCRIPTION OF THE INVENTION

Definitions:

For the purpose of describing the present invention, the following terms have these meanings: "Critical distance" means the distance separating the

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms <sup>15</sup> microbubbles and nanobubbles.

"O<sub>2</sub> emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than <sup>20</sup> 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance. 25

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O<sub>2</sub>. In the special dimensions of the invention, as explained in more detail in the following examples, O<sub>2</sub> forms bubbles which are too small to break the surface tension of the fluid. These bubbles <sup>40</sup> remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> 45 formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and 50 low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water 55 became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and 60 nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided

4

sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a  $\frac{1}{16}$  inch mesh marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2 shows a plan view of the assembled device. The  $O_2$ emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected 10 to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon® polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. **3** shows a block diagram of a timer control with anode **1**, cathode **2**, thermistor temperature sensor **3**, timer control circuit **4** and wire from a direct current power source **5**.

#### EXAMPLE 2

#### Measurement of O<sub>2</sub> Bubbles

Attempts were made to measure the diameter of the  $O_2$  bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 65 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outlines by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the

5

scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size 5 range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the 15 oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch 35 diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more 45 convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses

TABLE I

Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts	
Bait keeper	5	6	0.090	0.060	0.36	•
Livewell	32	12	0.180	0.120	1.44	
OEM 2 inch	10	12	0.210	0.120	1.44	~ ~
Bait store	70	12	0.180	0.180	2.16	55
Double cycle	2	12	0.180	0.180	2.16	
OEM 3 inch	50	12	0.500	0.265	3.48	
OEM 4 inch	80	12	0.980	0.410	4.92	
Water pail	2	24	1.200	1.200	28.80	
Plate	250	12	5.000	2.500	30.00	
						. 60

#### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An  $O_2$  emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum

6

anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

10	cathode screen nylon spacer anode grid nylon spacer cathode screen	0.045 inches thick 0.053 inches thick 0.035 inches thick 0.053 inches thick 0.045 inches thick	
	cathode screen	0.045 menes tines,	

for an overall emitter thickness of 0.231 inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, cathode, ode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

I claim:

1. An emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other.

2. The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

**3**. The emitter of claim **1** wherein the anode is platinum and iridium oxide on a support.

4. The emitter of claim 1 wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

5. The critical distance of claim 1 which is 0.005 to 0.140 inches.

**6**. The critical distance of claim  $\mathbf{1}$  which is 0.045 to 0.060 inches.

**7**. A method for lowering the biologic oxygen demand of polluted water comprising passing the polluted water through a vessel containing the emitter of claim **1**.

8. The product of claim  $\tilde{1}$  wherein the water is supersaturated with oxygen and of an approximately neutral pH.

 An emitter for electrolytic generation of microbubbles of oxygen comprising a plurality of anodes separated at a <sup>50</sup> critical distance from a plurality of cathodes and a power

source all in electrical communication with each other. **10.** A method for keeping aquatic animals emitter alive

comprising inserting the emitter of claim 1 or claim 9 into the aquatic medium of the aquatic animals. 11. The method of claim 8 wherein the aquatic animal is

**11.** The method of claim **8** wherein the aquatic animal is a fish.

12. The method of claim 8 wherein the aquatic animal is a shrimp.

13. An emitter for electrolytic generation of microbubbles of oxygen comprising a platinum-iridium oxide anode on a titanium support separated at a critical distance of from 0.045 inches to 0.060 inches from a stainless steel screen  $V_{16}$ inch thick cathode all in electrical communication with a battery.

14. The emitter of claims 1, 9 or 13 further comprising a timer control.

\* \* \* \* \*

**JA66** 



## (12) United States Patent Senkiw

#### (54) FLOW-THROUGH OXYGENATOR

- (75) Inventor: James Andrew Senkiw, Minneapolis, MN (US)
- (73) Assignee: Aqua Innovations, Inc., Minnetonka, MN (US)
- (\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 7 days.

This patent is subject to a terminal disclaimer.

- (21) Appl. No.: 10/732,326
- (22) Filed: Dec. 10, 2003

#### Prior Publication Data

US 2004/0118701 A1 Jun. 24, 2004

#### **Related U.S. Application Data**

- (63) Continuation-in-part of application No. 10/372,017, filed on Feb. 21, 2003, now Pat. No. 6,689,262.
- (60) Provisional application No. 60/358,534, filed on Feb. 22, 2002.
- (51) Int. Cl.

(65)

C25B 1/02	(2006.01)
C25B 1/04	(2006.01)
C02F 1/00	(2006.01)

- (58) Field of Classification Search ....... 205/628–639, 205/742; 204/242, 245, 275.1, 278.5, 290.1, 204/232, 278, 286.1, 554, 660; 210/243, 210/748

See application file for complete search history.

 (10) Patent No.:
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 (45) Date of Patent:
 \*Jul. 8, 2008

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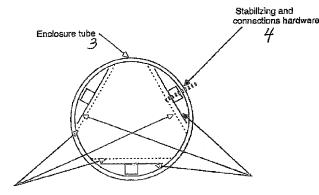
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Primary Examiner—Roy King Assistant Examiner—Lois L. Zheng (74) Attorney, Agent, or Firm—Patterson, Thuente, Skaar & Christensen, P.A.

#### (57) ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

#### 17 Claims, 8 Drawing Sheets



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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 70 of 1320

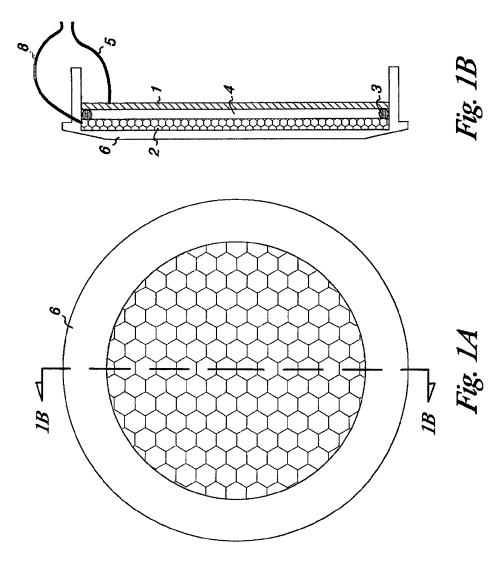
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Sheet 1 of 8

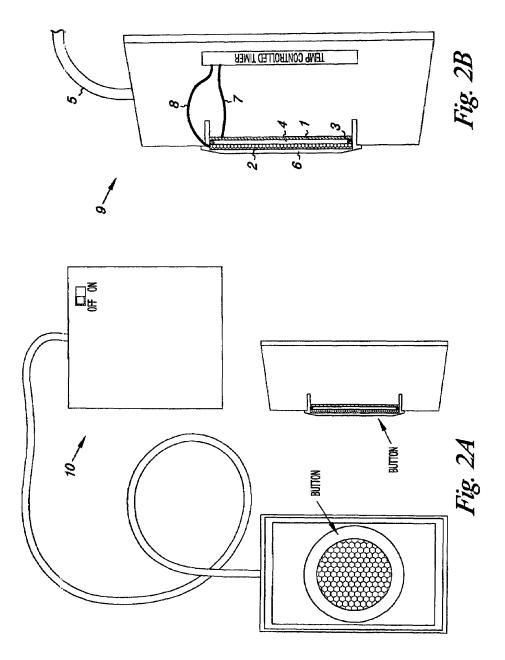
US 7,396,441 B2





Sheet 2 of 8

US 7,396,441 B2



U.S. Patent

Jul. 8, 2008

Sheet 3 of 8

US 7,396,441 B2

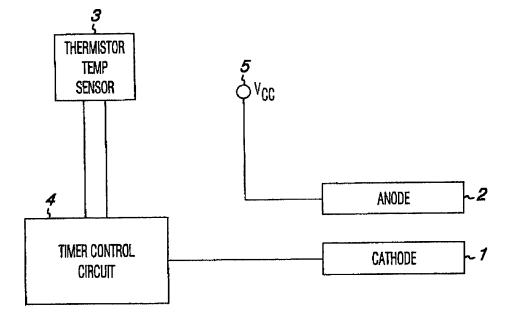
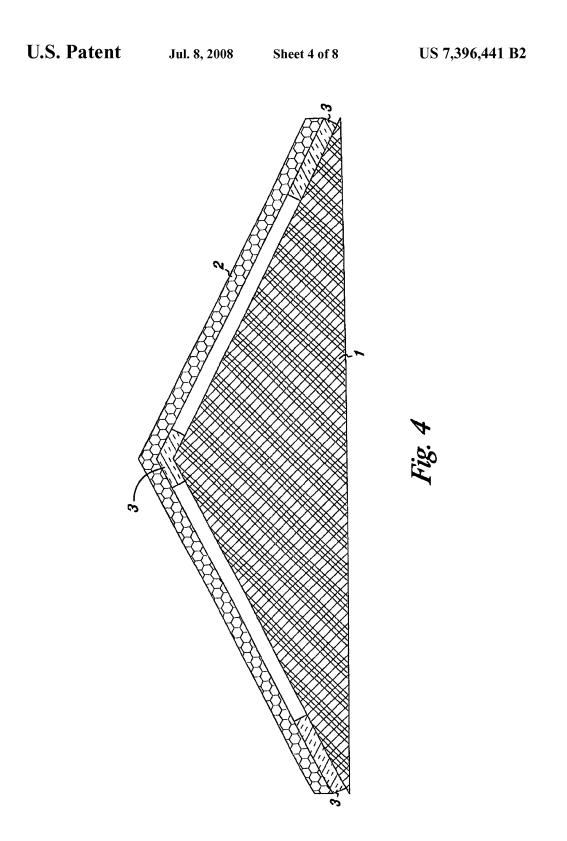


Fig. 3

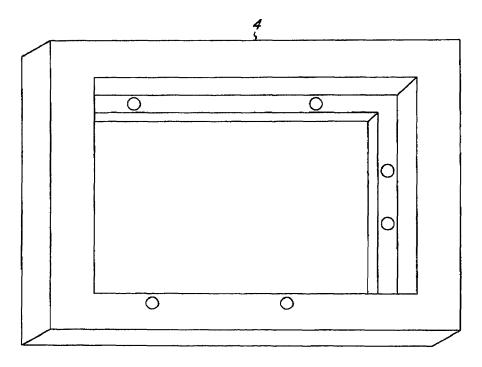


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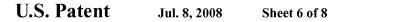
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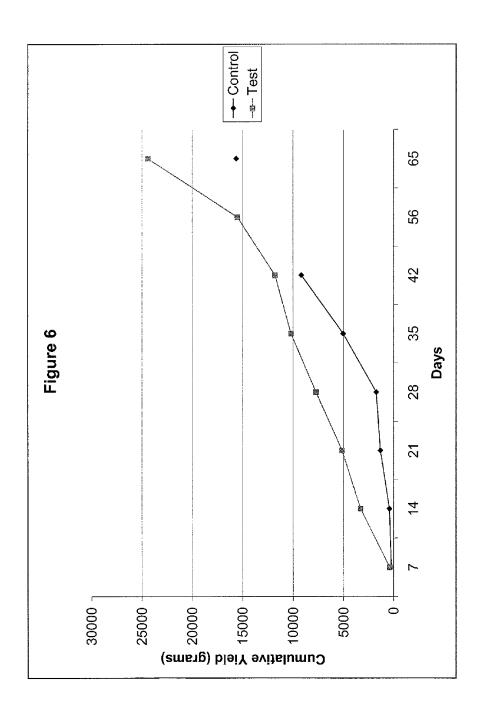
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US 7,396,441 B2



*Fig.* 5*B* 





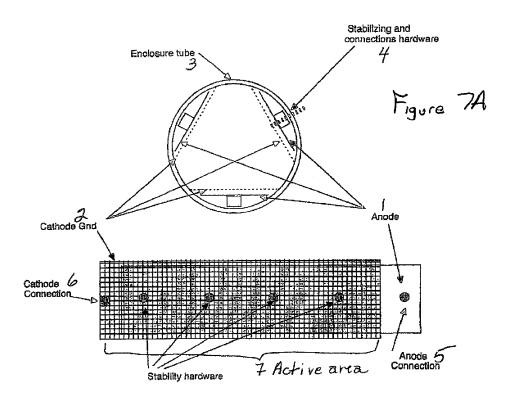
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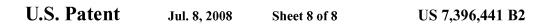
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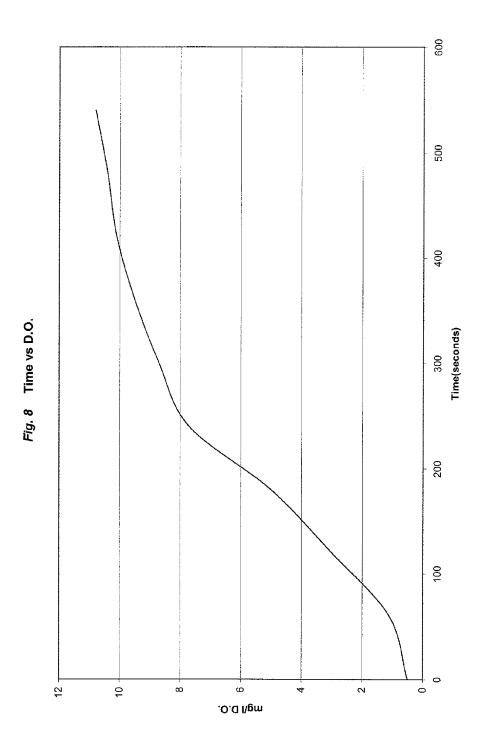
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## 3 Element Flow Through Oxygenation Chamber



Depending on requirements tube can contain 1 2 3 4 or more elements.





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#### 1 FLOW-THROUGH OXYGENATOR

#### RELATED APPLICATIONS

This application is a continuation-in-part of U.S. patent 5 application Ser. No. 10/372,017, filed on Feb. 21, 2003, now U.S. Pat. No. 6,689,262, issued Feb. 10, 2004, which claims priority to U.S. Provisional Patent Application No. 60/358, 534, filed Feb. 22, 2002.

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applica-25 tions such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollut- 35 ants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or 4 by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the 50 bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534, 143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a 55 device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure 60 oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is 65 produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE ANODE: $2H_2O \rightarrow O_2 + 4H^* + 4e^-$ NET REACTION: $6H_2O \rightarrow 0Q^+ + 4H^* + 2H_2 + O_2$		
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286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen. The electrodes may be a metal or oxide of at least one metal

selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium

3

and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a  $\frac{1}{16}$  inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to <sup>10</sup> various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held. <sup>15</sup>

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

#### DESCRIPTION OF THE DRAWINGS

FIG. 1A is a plan view of an  $O_2$  emitter of the invention. FIG. 1B is a section view of the  $O_2$  emitter of FIG. 1A taken

at line 1B-1B of FIG. 1A.

FIG. **2**A is a plan view of an assembled O<sub>2</sub> emitting device. <sup>45</sup> FIG. **2**B is a perspective view of the assembled O2 emitting device of FIG. **2**A.

FIG. 3 is a diagram of the electronic controls of the  $O_2$  emitter.

FIG. 4 shows a funnel or pyramid variation of the  $\mathrm{O}_2$  emitter.

FIG. **5** shows a multilayer sandwich  $O_2$  emitter.

FIG. 6 shows the yield of tomato plants watered with

superoxygenated water. 5: FIG. 7A is a cross section showing arrangement of three plate electrodes.

FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water. <sup>60</sup>

DETAILED DESCRIPTION OF THE INVENTION

#### Definitions:

For the purpose of describing the present invention, the following terms have these meanings:

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O<sub>2</sub> emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIGS. 1A, 1B, 2A and 2B, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of itianium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a {fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen.
The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source

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through a connection point 5. FIG. 2A shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical 5 distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the 10 spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon-.RTM. polymer or other plastic. Because of the criticality of 1 the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain 30 duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer 35 control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### EXAMPLE 2

#### Measurement of O2 Bubbles

Attempts were made to measure the diameter of the  $O_2$ bubbles emitted by the device of Example 1. In the case of 45 particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, 50 backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was 55 enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the 60 scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so 65 monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to

6

increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

TABLE I

Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts
Bait keeper	5	6	0.090	0.060	0.36
Livewell	32	12	0.180	0.120	1.44
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5,000	2.500	30.00

#### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An  $O_2$  emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

cathode screen	0.045 inches thick
nylon spacer	0.053 inches thick
anode grid	0.035 inches thick
nylon spacer	0.053 inches thick
cathode screen	0.045 inches thick,
	for an overall emitter thickness
	of 0.231 inches.

7

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

#### EXAMPLE 5

# Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthe- 25 sis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass  $_{40}$ of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

#### A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two  $^{\rm 45}$ tomato plants. Circulation protocols were identical except that the 21/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes 50 prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor 60 environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

#### B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the appli8

cation of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen. Tomato seeds (Burpee "Big Boy") were planted in oneinch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week.

After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

-		-	**
TΑ	$_{\rm BL}$	Æ	Π

Week of:	tomate eight	ol, grams oes from plants/ tive total	tomatc seven	grams es from plants/ tive total
July 27	240		400	
August 3	180	420	2910	3310
August 10	905	1325	1830	5140
August 17	410	1735	2590	7730
August 24	3300	5035	2470	10200
August 31	4150	9175	1580	11780
September 15	not w	eighed	3710	15490
Final Harvest September 24	6435	15620	8895	2438:

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. **6** shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

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#### 9 EXAMPLE 6

#### Flow-through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural 5 uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at  $120^{\circ}$  angles to 10 each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any <sup>20</sup> of the emitters shown in FIG. **4** or **5**, or can design other embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows <sup>25</sup> several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2° C. but the flowing water cooled slightly to 11 or 11.5° C. Without undue experimen-<sup>30</sup> tation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE III

MODEL	ACTIVE ELECTRODE AREA, SQ. IN.	VOLT- AGE	CUR- RENT, AMPS	FLOW RATE GAL/ MINUTE	DO OF* SAMPLE AT ONE MINUTE
2-inch "T"	2	28.3	0.7	12	N/A
3-inch "T"	3	28.3	1.75	12	N/A
2-plate Tube	20	28.3	9.1	12	8.4
3-Plate tube	30	28.3	12.8	12	9.6

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The one-minute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will 50 be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the sort field, this technique is especially useful in and climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly. 65

It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more

#### 10

continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

#### EXAMPLE 7

#### Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

I claim:

1. A flow through oxygenator comprising:

- a fluid conduit having a fluid inlet and a fluid outlet fluidly connected with a conduit lumen;
- an oxygen emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, the oxygen emitter including three matched sets of anodes and cathodes wherein the matched sets of anodes and cathodes are mounted to stabilizing hardware such that the oxygen emitter is positioned within the conduit lumen and each matched set resides at a 120° angle to the adjacent matched sets; and

a power source in electrical communication with the oxygen emitter.

2. The flow through oxygenator of claim 1, wherein each anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and each cathode is a metal or metallic oxide or a combination of a metal and a metallic 40 oxide.

**3**. The flow through oxygenator of claim **1**, wherein the anode and cathode within each matched set are separated by a spacer such to maintain a gap of 0.005 to 0.140 inches between the anode and cathode.

**4**. The flow through oxygenator of claim **3**, wherein the gap is 0.045 to 0.060 inches.

5. The flow though oxygenator of claim 1 wherein each anode is platinum and iridium oxide on a support and each cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

6. The flow though oxygenator of claim 1, wherein the power source is electrically connected to the stabilizing hardware for powering the plurality of matched sets of anodes and cathodes.

**7**. The flow through oxygenator of claim **1**, wherein the plurality of matched sets of anodes and cathodes are attached to the stabilizing hardware with the anodes proximate a conduit wall and the cathodes proximate a conduit center.

**8**. The flow through oxygenator of claim **1**, wherein the plurality of matched sets of anodes and cathodes define plates positioned parallel to a flow axis of the conduit lumen.

**9**. The flow through oxygenator of claim **1**, wherein each cathode comprises a mesh screen.

10. The flow through oxygenator of claim 1, further com-65 prising:

a controller selectively operating the power source, such that the power source supplies power to the plurality of

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA81** 

11

matched sets of anodes and cathodes when the aqueous medium is flowing through the conduit lumen and withholds power when the aqueous medium is not flowing through the conduit lumen.

11. The flow through oxygenator of claim 1, wherein the 5 oxygen emitter is sized to generate oxygen sufficient to form a supersaturated aqueous medium.

12. The flow through oxygenator of claim 1, wherein the aqueous medium is water.

13. The flow through oxygenator of claim 12, wherein the  $^{10}$ oxygen emitter is sized to generate oxygen sufficient to form superoxygenated water.

14. The flow through oxygenator of claim 1, wherein the fluid conduit is a watering hose.

15 15. The flow through oxygenator of claim 1, wherein the fluid conduit is a hydroponic circulating system.

#### 12

16. A flow through oxygenator comprising: a watering hose having a hose lumen; and

an oxygen emitter operably mounted within the hose lumen, the oxygen emitter including three matched sets of anodes and cathodes mounted to stabilizing hardware such that each matched set resides at a 120° angle to the adjacent matched sets.

17. A flow though oxygenator comprising:

- a hydroponic circulating system having a circulating lumen; and
- an oxygen emitter operably mounted within the circulating lumen, the oxygen emitter including three matched sets of anodes and cathodes mounted to stabilizing hardware such that each matched set resides at a 120° angle to the adjacent matched sets.

\* \* \* \* \*

CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 85 of 1320

(56)



US 7,670,495 B2

## (12) United States Patent Senkiw

#### (54) FLOW-THROUGH OXYGENATOR

- (75) Inventor: James Andrew Senkiw, Minneapolis, MN (US)
- Oxygenator Water Technologies, Inc., (73)Assignee: Minnetonka, MN (US)
- (\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

- (21) Appl. No.: 12/023,431
- (22) Filed: Jan. 31, 2008

#### (65) **Prior Publication Data**

US 2008/0179259 A1 Jul. 31, 2008

#### **Related U.S. Application Data**

- (60) Division of application No. 10/732,326, filed on Dec. 10, 2003, now Pat. No. 7,396,441, which is a continuation-in-part of application No. 10/372,017, filed on Feb. 21, 2003, now Pat. No. 6,689,262.
- (60) Provisional application No. 60/358,534, filed on Feb. 22, 2002
- (51) Int. Cl.

C02F 1/48	(2006.01)
C02F 1/00	(2006.01)
C25B 1/02	(2006.01)
C25B 1/04	(2006.01)

- U.S. Cl. ..... 210/748; 210/600; 210/243; (52)204/245; 204/232; 205/628
- Field of Classification Search ...... 210/748, (58)210/600, 243; 204/278, 242, 243, 275.1, 204/232, 286.1, 554, 660; 205/633-638 See application file for complete search history.

\*Mar. 2, 2010

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(45) Date of Patent:

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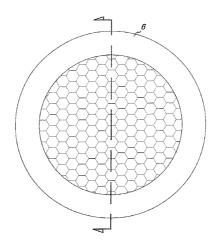
Primary Examiner-Walter D Griffin

Assistant Examiner-Cameron J Allen (74) Attorney, Agent, or Firm-Patterson, Thuente, Skaar & Christensen, P.A.

#### (57)ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

#### 12 Claims, 8 Drawing Sheets



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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 86 of 1320

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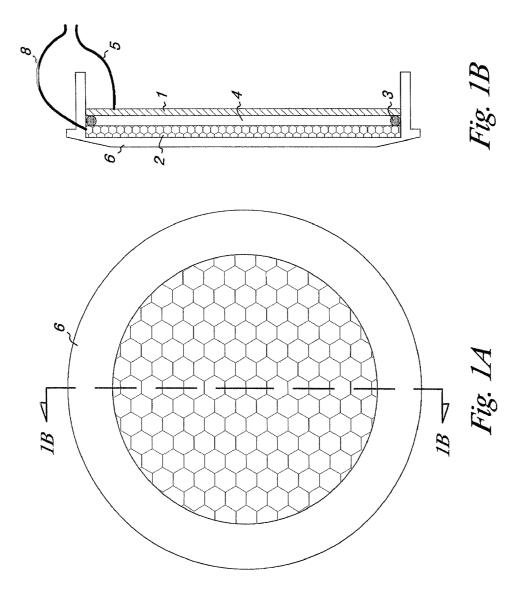
## US 7,670,495 B2

Page 2



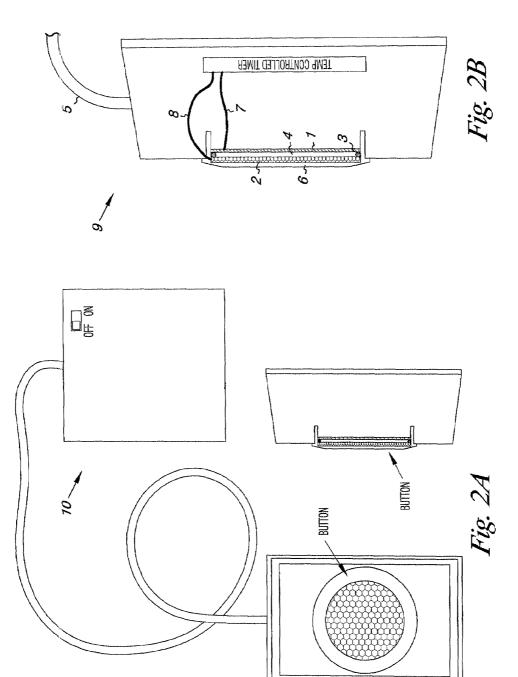
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US 7,670,495 B2





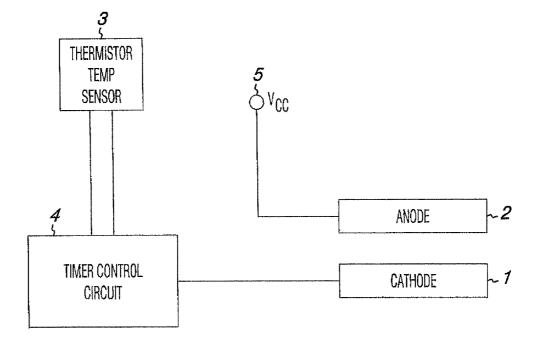
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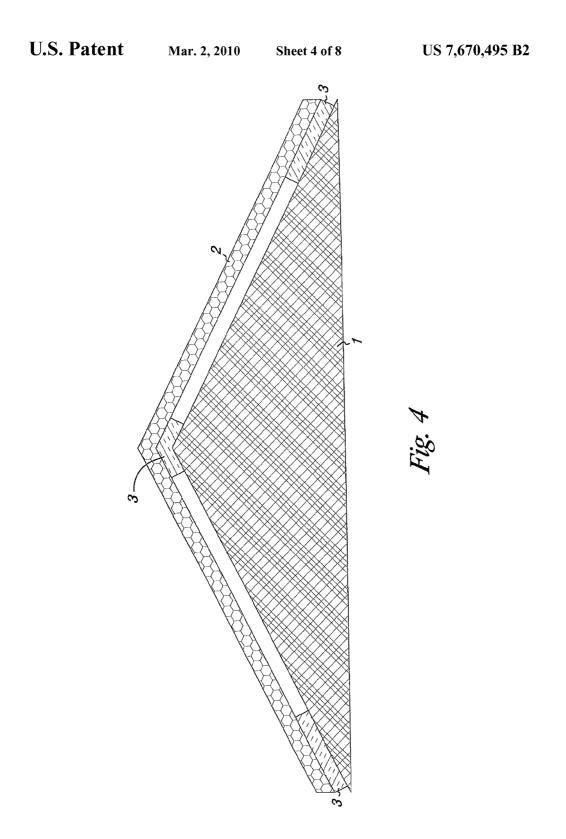


Sheet 3 of 8

US 7,670,495 B2



*Fig. 3* 



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Mar. 2, 2010

Sheet 5 of 8

US 7,670,495 B2

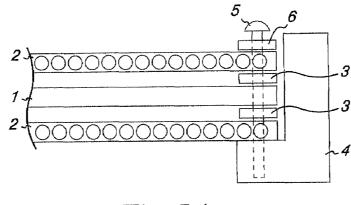


Fig. 5A

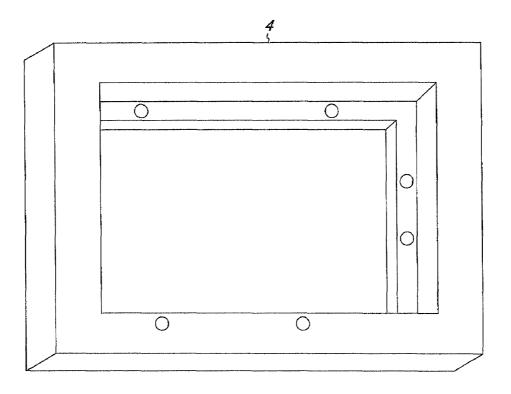
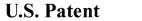
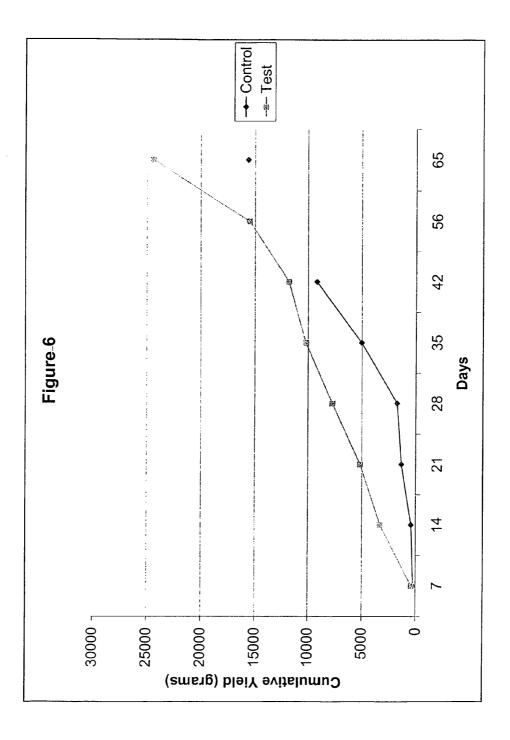


Fig. 5B

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Sheet 6 of 8

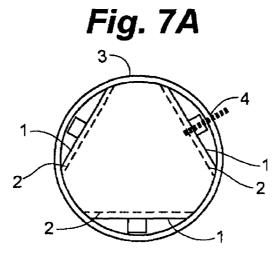


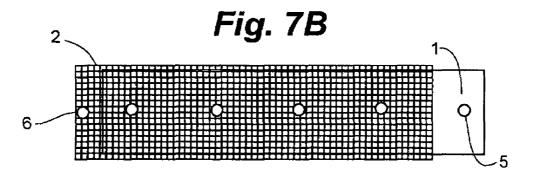
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Sheet 7 of 8

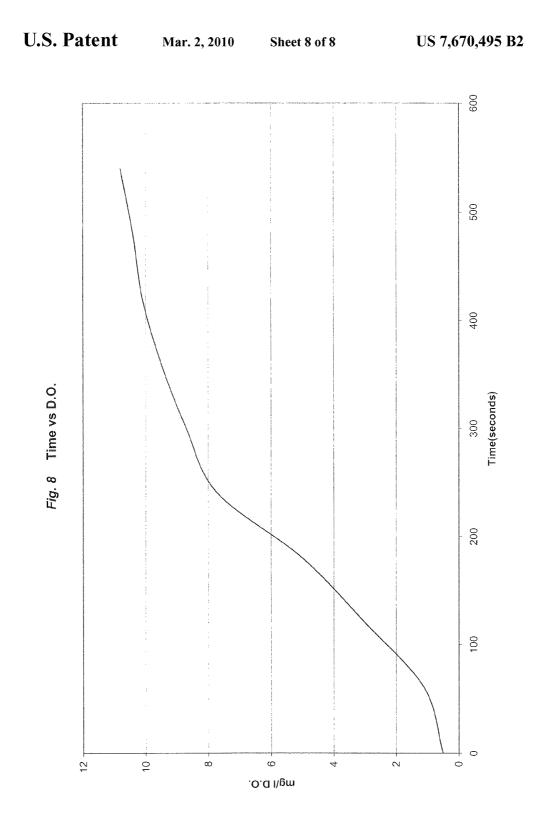
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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA91** 



#### 1 FLOW-THROUGH OXYGENATOR

#### RELATED APPLICATIONS

This application is a division of application Ser. No. <sup>5</sup> 10/732,326 filed Dec. 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by <sup>10</sup> reference.

#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of <sup>15</sup> microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water <sup>20</sup> treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely  $_{40}$ affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this 50 is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534, 55 143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high 60 pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied 65 across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a

2

battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE: AT THE ANODE: NET REACTION:	$\begin{array}{l} 4\mathrm{H}_{2}\mathrm{O}+4\mathrm{e}^{-}\rightarrow4\mathrm{OH}^{-}+2\mathrm{H}_{2}\\ 2\mathrm{H}_{2}\mathrm{O}\rightarrow\mathrm{O}_{2}+4\mathrm{H}^{+}+4\mathrm{e}^{-}\\ 6\mathrm{H}_{2}\mathrm{O}\rightarrow4\mathrm{OH}^{-}+4\mathrm{H}^{+}+2\mathrm{H}_{2}+\mathrm{O}_{2} \end{array}$
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286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

#### SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

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20

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a {fraction (1/16)} inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches

Models of different size are provided to be applicable to 15 various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the  $O_2$  emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the  $O_2$ emitter

FIG. 4 shows a funnel or pyramid variation of the  $O_2^{-50}$ emitter

FIG. 5 shows a multilayer sandwich  $O_2$  emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-55 through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source

FIG. 8 is a graph showing the oxygenation of waste water. <sup>60</sup>

DETAILED DESCRIPTION OF THE INVENTION

#### Definitions

For the purpose of describing the present invention, the following terms have these meanings:

4

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

'Critical distance'' means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O<sub>2</sub> emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals. "Microbubble" means a bubble with a diameter less than 50 microns

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

'Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples, O2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button 45 emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode **2** is a (fraction  $(\frac{1}{16})$  inch mesh (size 8 mesh) marine stainless steel screen. The anode and 65 cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a

connection point 5. FIG. 2 shows a plan view of the assembled device. The O2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of 5 sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to  $\ ^{10}$ 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon®, polymer or other plastic. Because of the criticality of the space distance, it is preferable 1 to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

-5

In operation, a small device with an  $O_2$  emitter 1.485 inches <sup>20</sup> in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises <sup>25</sup> a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### EXAMPLE 2

#### Measurement of O2 Bubbles

Attempts were made to measure the diameter of the  $O_2$ bubbles emitted by the device of Example 1. In the case of  $_{45}$ particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the 60 scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so 65 monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to

6

increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

#### Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles  $(H_2)$  arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial  $\frac{1}{40}$ use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

TABLE I

Emitter Model	Gallons	Volts	Amps Max.	Ave	Watts
Bait keeper	5	6	0.090	0.060	0.36
Livewell	32	12	0.180	0.120	1.44
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5,000	2,500	30.00

#### EXAMPLE 4

#### Multilayer Sandwich O2 Emitter

An  $O_2$  emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

cathode screen 0.045 inches thick,		cathode screen nylon spacer anode grid nylon spacer cathode screen	0.045 inches thick 0.053 inches thick 0.035 inches thick 0.053 inches thick 0.045 inches thick,	
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7

for an overall emitter thickness of 0.231 inches thick inches. 10

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

#### EXAMPLE 5

# Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthe- 25 sis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass  $_{40}$ of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

#### A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two <sup>45</sup> tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor 60 environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed

#### B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the appli8

cation of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in oneinch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

	DI		
LA		ΔD2	

45	Week of:	Control, g tomatoes eight pla cumulative	from nts/	Test, grams tomatoes from seven plants/ cumulative total		
	July 27	240		400		
	August 3	180	420	2910	3310	
	August 10	905	1325	1830	5140	
	August 17	410	1735	2590	7730	
50	August 24	3300	5035	2470	10200	
	August 31	4150	9175	1580	11780	
	September 15	not weighed		3710	15490	
	Final Harvest September 24	6435	15620	8895	24385	

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

Page 99

## US 7,670,495 B2

#### **9** EXAMPLE 6

#### Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural 5 uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at  $120^{\circ}$  angles to 10 each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any 20 of the emitters shown in FIG. **4** or **5**, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows 25 several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2° C. but the flowing water cooled slightly to 11 or 11.5° C. Without undue experimen-30 tation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

#### 10

continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

#### EXAMPLE 7

#### Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. **8**, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

The invention claimed is:

- 1. A method for treating waste water comprising;
- providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other,

placing the emitter within a conduit; and

passing waste water through the conduit.

**2**. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive

	TABLE III						
MODEL	ACTIVE ELECTRODE AREA, SQ.IN.	VOLTAGE	CURRENT, AMPS.	FLOW RATE GAL/MINUTE	DO OF* SAMPLE AT ONE MINUT		
2-Inch "T"	2	28.3	0.72	12	N/A		
3-inch "T"	3	28.3	1.75	12	N/A		
2-plate Tube	20	28.3	9.1	12	8.4		
3-Plate tube	30	28.3	12.8	12	9.6		

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The one-minute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly. 65

It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breading the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

**3**. The emitter of claim **2**, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

**4**. The emitter of claim **2**, wherein the anode is platinum and iridium oxide on a support.

5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic <sub>65</sub> oxide.

**6**. The emitter of claim **2**, wherein the critical distance is 0.005 to 0.060 inches.

11

7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes. 8. A method for oxygenating a non-native habitat for tem-

porarily keeping aquatic animals, comprising: inserting the emitter of claim 2 into the aqueous medium, 5 the non-native habitat comprising an aquarium, a bait bucket or a live well.

**9**. A method for lowering the biologic oxygen demand of polluted water comprising:

passing the polluted water through a vessel containing the 10 emitter of claim **2**.

#### 12

**10.** A supersaturated aqueous product formed with the emitter of claim **2**, the supersaturated aqueous product having an approximately neutral pH.

11. The emitter of claim 2, further comprising a timer control.

**12**. The emitter of claim **2**, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.

\* \* \* \* \*

## UNITED STATES PATENT AND TRADEMARK OFFICE CERTIFICATE OF CORRECTION

 PATENT NO.
 : 7,670,495 B2

 APPLICATION NO.
 : 12/023431

 DATED
 : March 2, 2010

 INVENTOR(S)
 : Senkiw

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

<u>Column 10, Line 55</u>: Delete "breading" and insert --breaking--.

Signed and Sealed this

First Day of June, 2010

and J. Kappos

David J. Kappos Director of the United States Patent and Trademark Office

## PATENT ASSIGNMENT

## Electronic Version v1.1 Stylesheet Version v1.1

	SUBMISSION TYPE:		WASSIGNMENT		NEW ASSIGNMENT		
NATURE OF CONVEYANCE:			LICENSE				
CONVEYING PART	Y DATA						
		Name		Execution Date			
Oxygenator Water Technologies, Inc.				10/04/2012			
Aqua Innovations Incorporated				10/04/2012			
RECEIVING PARTY	DATA						
Name: Roy H Lecy							
Street Address: 2640 North Saunders Lake Drive							
City:	Minnetrista						
State/Country:	MINNESOTA						
Postal Code: 55364							
		89262					
Property Type			Number	]			
		96441	441				
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 103 of 1320

Signature:	/nathanmbrandenburg/		
Date:	04/30/2013		
	This document serves as an Oath/Declaration (37 CFR 1.63).		
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## License Agreement

THIS AGREEMENT ("<u>Agreement</u>") is entered into this 30th day of July, 2008 (the "<u>Effective Date</u>"), by and between Oxygenator Water Technologies, Inc., a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensor</u>") and Aqua Innovations, Inc. a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensee</u>", and Licensor and Licensee each a "<u>Party</u>" and together the "<u>Parties</u>"). Initially capitalized terms defined in this Agreement shall have the meaning ascribed to them respectively herein.

## WITNESSETH:

LICENSOR owns the technology for which patents have been issued and are pending with respect to electrolytic hydrolysis of water to increase its dissolved oxygen content. A more complete description of said technology, together with a description of the patents issued and currently pending for said technology, is set forth in Article 1 below and in Exhibit "A" attached hereto.

LICENSOR anticipates and intends that it will make additional discoveries and improvements to said technology, some of which may be patentable.

It is further anticipated by the parties that LICENSOR may make improvements to said technology and additional discoveries concerning other applications for said technology.

The parties desire that LICENSOR grant a perpetual, exclusive license to LICENSEE to develop and sell throughout the world certain products utilizing the technology LICENSOR has developed and may in the future develop, all according to the terms and conditions set forth in this Agreement.

The parties further desire that LICENSOR will retain the complete and entire right to develop and sell throughout the world in markets not licensed to LICENSEE hereunder products utilizing the technology LICENSOR has developed and may in the future develop or the technology that LICENSEE may develop in the future, also according to the terms and conditions set forth in this Agreement.

Thus, the parties have agreed to enter into a licensing arrangement by which each party will be entitled to benefit from the other party's patents, technology and know-how concerning electrolytic hydrolysis of water in the sale of products in certain markets.

**NOW, THEREFORE,** based on the foregoing and the mutual covenants and agreements herein contained, the parties hereby covenant and agree as follows:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA102** 

EXHIBIT "B"

## **LICENSEE Markets**

All worldwide markets for:

- Waste Water Treatment
- Medical Applications
- Sport Fishing
- Aqua Culture
- Horticulture (consumer and commercial)
- Hydroponics

Markets excluded from license agreement (including but not limited to):

- Water Treatment (all applications except waste water)
- Fermentation
- Desalination
- Human Nutrition
- Animal Nutrition

## ARTICLE 1 DEFINITIONS

When used in this Agreement, the following terms have the meanings set forth below unless a different and common meaning of the term is clearly indicated by the context, and variants and derivatives of the following terms shall have correlative meanings:

"Agreement" has the meaning set forth in the preamble.

"LICENSOR Documents" has the meaning set forth in Section 2.6.

"LICENSOR Improvements" means all developments LICENSOR may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable, and which are invented, developed, discovered or otherwise acquired by LICENSOR and which LICENSOR may lawfully communicate to LICENSEE.

"*LICENSOR Markets*" means all uses for the LICENSOR Technology and the LICENSEE Technology other than in the LICENSEE Markets.

"LICENSOR Patents" means all of LICENSOR's patents (whether issued to LICENSOR or controlled by license rights or otherwise and whether such rights are held alone or jointly with others, and patents pending now, or during the term of this Agreement, issued to LICENSOR (by any country) relating to the LICENSOR Technology, including, but not limited to, those patents and those patents pending described on Exhibit A and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSOR Products*" means any product manufactured and/or sold or distributed by LICENSOR or a sub licensee of LICENSOR under any claim contained in the LICENSEE Patents.

"LICENSOR Property" means LICENSOR Patents, LICENSOR Improvements and LICENSOR Technology.

"LICENSOR Technology" means LICENSOR's unpatented technology and information now existing and relating to, and embodying LICENSOR's experience in electrolytic hydrolysis of water. LICENSOR Technology shall include the technical information in all current and future manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSOR to LICENSEE during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSOR imparting the same directly or giving LICENSEE access to any of LICENSOR's production facilities.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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"Effective Date" has the meaning set forth in the preamble.

"LICENSEE Documents" has the meaning set forth in Section 2.7.

"LICENSEE Improvements" means all developments LICENSEE may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable and which are invented, developed, discovered or otherwise acquired by LICENSEE and which LICENSEE may lawfully communicate to LICENSOR.

"*LICENSEE Markets*" means those markets for Licensee Products as are described in Exhibit B attached hereto.

"LICENSEE Patents" means all of LICENSEE's patents (whether issued to LICENSEE or controlled by license rights or otherwise and whether such rights are held alone or jointly with others) which may after the effective date of this Agreement be issued (by any country) relating to electrolytic hydrolysis of water and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSEE Products*" means any product manufactured and/or sold or distributed to any party other than LICENSOR by LICENSEE or a sublicense of LICENSEE in conformity with the terms of this Agreement, including, but not limited to, any product which is based on any claim or thing contained in any LICENSOR Property.

"LICENSEE Property" means LICENSEE Patents, LICENSEE Improvements and LICENSEE Technology.

"LICENSEE Technology" means LICENSEE's unpatented technology and information which LICENSEE may develop relating to, and embodying LICENSEE's experience in, the manufacturing, the processing, quality control, and sale of the LICENSEE Products. LICENSEE Technology shall include the technical information in all manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSEE to LICENSOR during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSEE imparting the same directly or giving LICENSOR access to any of LICENSEE's production facilities.

"Territory" means the world,

## ARTICLE 2 MARKETS AND LICENSING

2.1. Exclusive Markets. The parties agree that unless properly terminated by LICENSOR pursuant to Section 5.1 below, LICENSEE will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSEE Markets in the Territory. The parties further agree that LICENSOR will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSOR Markets in the Territory. LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSOR Products in the LICENSOR Markets in the Territory. LICENSEE may not, directly or indirectly, distribute in any manner any product which competes with the LICENSEE Products in any manner nor may LICENSEE assist or have any interest in any third party distributing any such products through licensing or assignment of technology to any such third party or by any other means.

2.2. Exclusive License to LICENSEE. Subject to the terms and conditions of this Agreement, LICENSOR hereby confers upon LICENSEE the sole and exclusive license, with the right of sublicense, under the LICENSOR Property, to make, have made, use and sell the LICENSEE Products in the LICENSEE Markets in the Territory and to prevent infringement of the LICENSOR Patents, and to prevent unauthorized use and disclosure of the LICENSOR Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSEE Products.

2.3. Exclusive License to LICENSOR. Subject to the terms and conditions of this Agreement, LICENSEE hereby confers upon LICENSOR the sole and exclusive, royalty-free license, with the right of sublicense, under the LICENSEE Property, to make, have made, use and sell the LICENSOR Products in the LICENSOR Markets in the Territory and to prevent infringement of the LICENSEE Patents, and to prevent unauthorized use and disclosure of the LICENSEE Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSOR Products.

**2.4. Product Markings.** The Parties shall insure that all LICENSOR Products and all LICENSEE Products are marked with any applicable patent number and all labeling and other product information shall be marked in such manner as to conform with the patent laws and practices of the country of sale.

2.5 Transfer of Technology by LICENSOR. As promptly as practicable after the execution of this Agreement, LICENSOR shall deliver to LICENSEE all information concerning the LICENSOR Property. LICENSOR also promptly shall deliver to LICENSEE all future information it acquires concerning the LICENSOR Property. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSOR, shall be referred to as "LICENSOR Documents". LICENSEE shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSOR Documents in such manner as to maximize the value of the LICENSOR Patents, the LICENSOR Technology and the LICENSOR Improvements; without limiting the generality of the foregoing, LICENSEE shall, and shall cause its employees and

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA106** 

representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSOR Documents. Upon 10 days advance notice and at reasonable times, LICENSOR shall permit LICENSEE access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSOR to answer fully such questions as LICENSEE may have with a view to transferring the LICENSOR Property. Nothing in this Section 2.6 shall require LICENSOR to disclose to LICENSEE any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

## 2.6. Transfer of Technology by LICENSEE. As promptly as practicable after

LICENSEE develops, discovers or otherwise comes into possession of LICENSEE Patents, LICENSEE Improvements and/or LICENSEE Technology, LICENSEE shall deliver to LICENSOR all information concerning same. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSEE, shall be referred to as "LICENSEE Documents." LICENSOR shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSEE Documents in such manner as to maximize the value of the LICENSEE Patents, the LICENSEE Technology and the LICENSEE Improvements; without limiting the generality of the foregoing, LICENSOR shall, and shall cause its employees and representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSEE Documents. Upon 10 days advance notice and at reasonable times, LICENSEE shall permit LICENSOR access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSEE to answer fully such questions as LICENSOR may have with a view to transferring to LICENSOR the LICENSEE Property. Nothing in this Section 2.7 shall require LICENSEE to disclose to LICENSOR any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

2.7. Further Prosecution of Patents. LICENSOR will continue with the prompt prosecution of all pending patent applications filed by LICENSOR as detailed on Schedule "A", so long as it is commercially reasonable to do so, and LICENSOR will periodically advise LICENSEE of the status of such prosecutions. As soon as practical, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSOR Patents. In the event that LICENSEE files an application for a patent(s) covering electrolytic hydrolysis of water, LICENSEE will periodically advise LICENSOR of the status of the prosecution of any such patent. As soon as practical after any such application by LICENSEE, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSEE Patents. From the date of this Agreement, all expenses incurred in filing for and maintaining protection in those countries mutually agreed upon (other than expenses of prosecuting the original patent application in the first jurisdiction, which will be the responsibility of the Party filing the patent application) will be shared equally by the Parties. Either Party may seek protection in any country not mutually agreed upon by paying the full amount of the cost thereof. A party seeking such additional protection will receive the full cooperation of the other Party (other than in paying the expenses thereof) in protecting all patents in any such other country.

2.8. Additional Covenants. Each of LICENSOR and LICENSEE shall faithfully comply with their respective obligations under this Agreement and shall incorporate all terms and conditions required by this Agreement in any contracts with third parties to whom access to the LICENSOR Property or the LICENSEE Property, as the case may be, may (but only in accordance with this Agreement) be given. Each of LICENSOR and LICENSEE shall indemnify and hold harmless the other Party and its successors and assigns from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on such Party, and arising out of or in connection with or resulting from the marketing, sale or use of the imdemnifying Party's product(s), including any advertising or other promotional activities related thereto.

**2.9. Infringement Actions.** Neither LICENSOR nor LICENSEE will have any responsibility to the other Party for any damage or expense incurred by such other Party which arises from any action, claim or cause of action brought by any person as the result of any alleged patent infringement or trade secret misappropriation by reason of such other Party's manufacture, use or sale of any product under any of the licenses conferred hereby.

2.10. LICENSEE's Rights in Event of Third Party Infringement. LICENSEE shall have the right, in LICENSOR's name (if required by law, otherwise, in LICENSEE's name) but at LICENSEE's sole expense, to sue third parties in the LICENSEE Markets for infringements of the LICENSOR Patents and misappropriation of the LICENSOR Technology and unpatented LICENSOR Improvements, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. LICENSOR may, if it so elects, join in any such suit as a plaintiff. All damages, awards or settlement proceeds in such suit shall be LICENSEE's. If LICENSEE, after notice from LICENSOR of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSOR, in its own name (or, if required by law, in its and LICENSEE's name) and at its own expense, may sue therefore, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSOR shall promptly reimburse LICENSEE for said suitassociated direct expenses upon presentation of LICENSEE's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's.

2.11. LICENSOR's Rights in Event of Third Party Infringement. LICENSOR

shall have the right, in LICENSEE's name (if required by law, otherwise, in LICENSOR's name) but at LICENSOR's sole expense, to sue third parties in the LICENSOR Markets for infringements of the LICENSEE Patents and misappropriation of the LICENSEE Technology and unpatented LICENSEE Improvements, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSEE may, if it so elects, join in any such suit as a plaintiff. LICENSOR shall promptly reimburse LICENSEE for said suit-associated direct expenses upon presentation of LICENSEE's itemized

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA108** 

statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's. If LICENSOR, after notice from LICENSEE of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSEE, in its own name (or, if required by law, in its and LICENSOR's name) and at its own expense, may sue therefore, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSEE's.

**2.12. LICENSEE Royalty Payment.** None. License is granted without cost to LICENSEE.

# ARTICLE 3 INDEMNIFICATION

**3.1.** Indemnification by LICENSEE. LICENSEE shall indemnify and hold LICENSOR and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSOR arising out of or in connection with or resulting from the marketing, sale or use of the LICENSEE Products, including any advertising or other promotional activities related thereto. LICENSOR shall be an added insured party to LICENSEE's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSEE shall procure and have in place no later than the date on which LICENSEE first makes a delivery of any of the LICENSEE Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSOR is provided at least thirty (30) days advance written notice.

**3.2. Indemnification by LICENSOR.** LICENSOR shall indemnify and hold LICENSEE and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSEE arising out of or in connection with or resulting from the marketing, sale or use of the LICENSOR Products, including any advertising or other promotional activities related thereto. At such time, if any, as LICENSOR shall sell LICENSOR Products, LICENSOR shall add LICENSEE as an added insured party to LICENSOR's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSOR shall procure and have in place no later than the date on which LICENSOR first makes a delivery of any of the LICENSOR Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSEE is provided at least thirty (30) days advance written notice.

## ARTICLE 4 CONFIDENTIALITY

4.1. Restrictions on Use and Disclosure of LICENSOR Property by LICENSEE. LICENSEE shall use the LICENSOR Property in confidence and shall not

disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate: (a) if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSEE, or (b) upon the expiration of the obligation of LICENSEE under this Agreement to pay royalties to LICENSOR.

4.2. Restrictions on Use and Disclosure of LICENSEE Property by LICENSOR. LICENSOR shall use the LICENSEE Property in confidence and shall not disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSOR.

## 4.3. Employees; Third Parties Etc. In order to faithfully perform their

respective obligations under sections 4.1 and 4.2, the Parties shall limit access to the other Party's Property to only those of its officers, employees and agents who shall have a need to receive or have access to that portion, and then only for the purposes of the practice under the licenses conferred by this Agreement. Each Party will require any third party, to whom access may be authorized under this Agreement, to execute an appropriate confidentiality agreement.

4.4. Authorized Required Disclosures. Nothing in this Article 4 shall prevent a Party: (a) from complying (but only to the narrowest extent required by law and regulation and with due notice on any submissions to governmental agencies of the confidential or proprietary status of the information with a view toward restricting access to, and use or disclosure by, third parties) with reasonable requirements of governmental agencies to disclose information in order to receive legally required consents or permissions to manufacture or sell that Party's Products; or (b) from disclosing information under court order, but only after having made all reasonable efforts to secure the court's order to (i) limit production, use and disclosure practicable under the circumstances and (ii) hold all proceedings in camera with a sealed record.

# ARTICLE 5 RESOLUTION OF DISPUTES

All claims, disputes and other matters in question arising out of, or relating to, this Agreement or the performance thereof shall be submitted to, and determined by, arbitration if good faith negotiations between the parties do not resolve such claim, dispute or other matter within 60 days. Such arbitration shall proceed in accordance with the Commercial Arbitration Rules of the American Arbitration Association then pertaining (the "Rules"), insofar as such Rules are not inconsistent with the provisions expressly set forth in this Agreement, unless the parties mutually agree otherwise, and pursuant to the following procedures:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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(a) Notice of the demand for arbitration shall be filed in writing with the other Member and with the American Arbitration Association. Each Member shall appoint an arbitrator, and those party-appointed arbitrators shall appoint a third neutral arbitrator within 10 days. If the party-appointed arbitrators fail to appoint a third, neutral arbitrator within 10 days, such third, neutral arbitrator shall be appointed by the American Arbitration Association in accordance with the Rules. A determination by a majority of the panel shall be binding.

(b) Reasonable discovery shall be allowed in arbitration.

(c) All proceedings before the arbitrators shall be held in Minneapolis, Minnesota. The governing law shall be as specified in Section 8.1 below.

(d) The costs and fees of the arbitration, including attorneys' fees, shall be allocated by the arbitrators.

(e) The award rendered by the arbitrators shall be final and judgment may be entered in accordance with applicable law and in any court having jurisdiction thereof.

#### ARTICLE 6 NOTICES

**6.1.** Notices. All communications, demands, notices or objections required or permitted to be given or served under this Agreement shall be in writing and shall be deemed to have been duly given or made only if delivered in person, deposited in the United States mail, postage prepaid, for mailing by certified or registered mail, return receipt requested, or delivered by prepaid overnight courier service, addressed to the appropriate party as follows:

- If to LICENSOR: Richard Disrud, COO Aqua Innovations, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435
- If to LICENSEE: Jeffrey Brink, CEO Oxygenator Water Technology, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435

Either party may change its address by giving notice in writing, stating the new address, to the other Party as provided in the foregoing manner. Commencing on the tenth (10th) day after the giving of such notice, such newly designated address shall be such Party's address for the purpose of all communications, demands, notices or objections required or permitted to be given or served under this Agreement.

# ARTICLE 7 MISCELLANEOUS

**7.1. Governing Law; Court Proceedings.** The validity, performance, and all matters relating to the interpretation and effect of this Agreement shall be governed by the internal law in effect in the State of Minnesota without regard to principles of law (such as "conflicts of law") that might make the law of some other jurisdiction applicable. Without limiting the terms set forth in Article 6 with respect to the resolution of disputes, each Party agrees to the exclusive and irrevocable jurisdiction arising out of or in any way related to this Agreement which may be brought in a court of law and both parties agree that personal service from any such court may be effectively served upon a party at the respective addresses set forth in Section 7.1.

**7.2. Exhibits.** Exhibits, schedules and annexes referred to in this Agreement and attached hereto are incorporated herein in full by this reference as if each of such exhibits, schedules or annexes were set forth in the body of this Agreement and duly executed by the parties hereto.

**7.3. Additional Documents and Acts.** Each party agrees that it will use all reasonable efforts to take, or cause to be taken, all actions and to do, or cause to be done, all things necessary, proper or advisable, including, but not limited to, the execution of additional documents and instruments, to consummate, make effective and carry out the transactions contemplated by this Agreement.

7.4. Amendment, Modification or Waiver. No amendment, modification or waiver of any condition, provision or term of this Agreement shall be valid or of any effect unless made in writing, signed by the party or parties to be bound or its duly authorized representative and specifying with particularity the nature and extent of such amendment, modification or waiver. Any waiver by any party of a default of another party shall not affect or impair any right arising from any subsequent default.

**7.5. Severable Provisions.** Whenever possible, each provision of this Agreement will be interpreted in such manner as to be effective and valid under applicable law, but if any provision of this Agreement is held to be invalid, illegal or unenforceable under any applicable law or rule in any jurisdiction, such provision will be ineffective only to the extent of such invalidity, illegality, or unenforceability in such jurisdiction, without invalidating the remainder of this Agreement in such jurisdiction or any provision hereof in any other jurisdiction.

**7.6. Entire Agreement.** This Agreement contains the entire understanding of the parties hereto in respect of the transactions contemplated hereby and supersedes all prior agreements and understandings between the parties with respect to such subject matter.

7.7. Captions, Headings, Titles or References to Gender. All captions, headings

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

or titles in the paragraphs or sections of this Agreement are inserted for convenience of reference only and shall not constitute a part of this Agreement or as a limitation of the scope of the particular paragraphs or sections to which they apply. Where appropriate, the masculine gender may be read as the feminine gender or the neuter gender, the feminine gender may be read as the masculine gender or the neuter gender and the neuter gender may be read as the masculine gender or the feminine gender.

**7.8.** Counterparts. This Agreement may be executed in two (2) or more counterparts, each of which shall be considered one and the same Agreement and shall become effective when one or more counterparts have been signed by each of the parties and delivered to the other parties.

IN WITNESS WHEREOF, the parties have executed this Agreement on the date first written above.

AQUA INNOVATIONS, INC.

1.1.2.=

Dick Disrud its COO

**OXYGENATOR WATER TECHNOLOGIES, INC.** 

Jeff Brink its CEO

EXHIBIT "A"

# DESCRIPTION OF LICENSOR PATENTS AND PATENTS PENDING

# United States Patent Number: US 6,689,262 B2 Date of Patent: February 10, 2004 Name: Microbubbles of Oxygen Application Number: 10/372,017

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen.

United States Patent Number: US 7,396,441 B2 Publication Date: July 8, 2008 Name: Flow-Thru Oxygenator Application Number: 10/732,326

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen. A flow-through model for oxygenating flowing water. The use of supersaturated water for enhancing the growth of plants. Method of applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture. The treatment of waste water by raising the dissolved oxygen with the use of oxygen emitter.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 117 of 1320

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<ul> <li>Resources &amp; Public Notices</li> </ul>	Examiner Name:	ALLEN, CAMERON J	Location: 👁	ELECTRONIC
Patent Searches	Group Art Unit:	1797	Location Date:	-
abent Official Gazetbe 1 Search Patents & Applications 1 Search Biological Secuences	Confirmation Number:	7381	Earliest Publication No:	US 2008-0179259 A1
<sup>1</sup> Copies, Products & Services	Attorney Docket Number:	4056.020503	Earliest Publication Date:	07-31-2008
Diher	Class / Subclass:	210/748	Patent Number:	7,670,495
opyrights rademarks slidy 6 Law soorts	First Named Inventor:	James Andrew Senkiw , Minneapolis, MN (US)	Issue Date of Patent:	03-02-2010
	Title of Invention:	DOW-THDOIC	+ OXYGENATOR	

Call the Patent Electronic Business Center at (866) 217-9197 (toll free) or e-mail EBC@uspto.gov for specific questions about Patent Application Information Retrieval (PAIR).
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THIS PATENT ISSUED 3/2/2010. In DISCUSSIONS WITH PAUL HAMN, IT GIVES US BREAD COVERAGE.

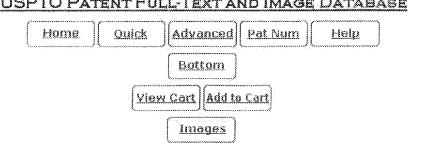
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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 118 of 1320

United States Patent: 7670495

Page 1 of 12



USPTO PATENT FULL-TEXT AND IMAGE DATABASE

**United States Patent** Senkiw

7,670,495 March 2, 2010

(1 of 1)

Flow-through oxygenator

#### Abstract

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flowthrough model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

Inventors: Senkiw; James Andrew (Minncapolis, MN) Assignce: Oxygenator Water Technologies, Inc. (Minnetonka, MN) Appl. No.: 12/023,431 Filed: January 31, 2008

#### **Related U.S. Patent Documents**

<b>Application</b> Number	<b>Filing Date</b>	Patent Number	<b>Issue Date</b>
10732326	Dec., 2003	7396441	
10372017	Feb., 2004	6689262	
60358534	Feb., 2002		

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Page 2 of 12

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Mohyuddin Mirza et al., "Effect of Oxygenated Water on the Growth & Biomass Development of Seedless Cucumbers and Tomato Seedlings under Greenhouse Conditions," Seair Diffusion Systems, 2003, 5 pages, www.seair.ca. cited by other.

Primary Examiner: Griffin; Walter D Assistant Examiner: Allen; Cameron J Attorney, Agent or Firm: Patterson, Thuente, Skaar & Christensen, P.A.

# Parent Case Text

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JA117

Page 3 of 12

RELATED APPLICATIONS

This application is a division of application Ser. No. 10/732,326 filed Dec. 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

Claims	
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The invention claimed is:

1. A method for treating waste water comprising; providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other, placing the emitter within a conduit; and passing waste water through the conduit.

2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breading the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.

5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.

7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.

8. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising: inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.

9. A method for lowering the biologic oxygen demand of polluted water comprising: passing the polluted water through a vessel containing the emitter of claim 2.

10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.

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#### Page 4 of 12

11. The emitter of claim 2, further comprising a timer control.

12. The emitter of claim 2, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.

Description

# FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

## BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

TABLE-US-00001 AT THE CATHODE: 4H.sub.2O + 4e.sup.- .fwdarw. 4OH.sup.- + 2H.sub.2 AT THE ANODE: 2H.sub.2O .fwdarw. O.sub.2 + 4H.sup.+ + 4e.sup.- NET REACTION: 6H.sub.2O .fwdarw. 4OH.sup.- + 4H.sup.+ ++ 2H.sub.2 + O.sub.2

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#### JA119

Page 5 of 12

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

# SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

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**JA120** 

Page 6 of 12

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

# BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the O.sub.2 emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the O.sub.2 emitter.

FIG. 4 shows a funnel or pyramid variation of the O.sub.2 emitter.

FIG. 5 shows a multilayer sandwich O.sub.2 emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

# DETAILED DESCRIPTION OF THE INVENTION

Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O.sub.2 emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O.sub.2. In the special dimensions of the invention, as explained in more detail in the following examples, O.sub.2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H.sub.2 formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

## EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 8 of 12

shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon.RTM., polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an O.sub.2 emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### **EXAMPLE 2**

#### Measurement of O.sub.2 Bubbles

Attempts were made to measure the diameter of the O.sub.2 bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed O.sub.2 bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

## EXAMPLE 3

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Page 9 of 12

Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H.sub.2) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

 TABLE-US-00002 TABLE I Emitter Model Gallons Volts Amps Max. Ave Watts Bait keeper 5 6 0.090
 0.060 0.36 Livewell 32 12 0.180 0.120 1.44 OEM 2 inch 10 12 0.210 0.120 1.44 Bait store 70 12 0.180

 0.180 2.16 Double cycle 2 12 0.180 0.180 2.16 OEM 3 inch 50 12 0.500 0.265 3.48 OEM 4 inch 80 12
 0.980 0.410 4.92 Water pail 2 24 1.200 1.200 28.80 Plate 250 12 5.000 2.500 30.00

**EXAMPLE 4** 

Multilayer Sandwich O.sub.2 Emitter

An O.sub.2 emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

TABLE-US-00003 cathode screen 0.045 inches thick nylon spacer 0.053 inches thick anode grid 0.035 inches thick nylon spacer 0.053 inches thick cathode screen 0.045 inches thick,

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

**EXAMPLE 5** 

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Page 10 of 12

Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in one-inch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 11 of 12

half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

TABLE-US-00004 TABLE II Control, grams Test, grams tomatoes from tomatoes from eight plants/ seven plants/ Week of: cumulative total cumulative total July 27 240 400 August 3 180 420 2910 3310 August 10 905 1325 1830 5140 August 17 410 1735 2590 7730 August 24 3300 5035 2470 10200 August 31 4150 9175 1580 11780 September 15 not weighed 3710 15490 Final Harvest 6435 15620 8895 24385 September 24

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

#### **EXAMPLE 6**

Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120.degree. angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2.degree. C. but the flowing water cooled slightly to 11 or 11.5.degree. C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE-US-00005 TABLE III ACTIVE DO OF\* ELECTRODE CURRENT, FLOW RATE SAMPLE AT MODEL AREA, SQ.IN. VOLTAGE AMPS. GAL/MINUTE ONE MINUTE 2-Inch "T" 2 28.3 0.72 12 N/A 3-inch "T" 3 28.3 1.75 12 N/A 2-plate Tube 20 28.3 9.1 12 8.4 3-Plate tube 30 28.3 12.8 12 9.6

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**JA126** 

Page 12 of 12

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The oneminute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

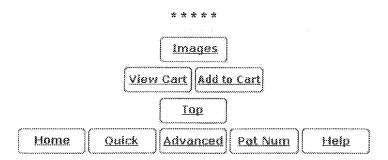
It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

EXAMPLE 7

Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.



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JA127

#### AGREEMENT OF STRICT FORECLOSURE

THIS AGREEMENT OF STRICT FORECLOSURE (the "<u>Agreement</u>") is made, entered into and effective as of October 4, 2012 (the "<u>Effective Date</u>"), by and between AQUA INNOVATIONS INCORPORATED ("<u>Debtor</u>"), a Minnesota corporation, and ROY H. LECY ("<u>Secured Party</u>"), who holds a certain security interest in the assets of Debtor as set forth below as security under a certain Promissory Note dated December 1, 2006, executed by Debtor in favor of Secured Party in the principal amount of Two Hundred Eighteen Thousand Eight Hundred Twenty-Seven and 29/100 Dollars (\$218,827.49) (the "<u>Note</u>"). Either Debtor or Secured Party may be individually referred to herein as a "<u>party</u>" or collectively as the "<u>parties</u>."

# RECITALS

WHEREAS, Secured Party is a shareholder of Debtor who holds nine hundred twentyeight thousand three hundred thirty-four (928,334) shares of Debtor's common stock and twentysix thousand six hundred sixty-seven (26,667) shares of Debtor's Preferred Series A stock; and

WHEREAS, Secured Party loaned Debtor various amounts of money over the course of several years, as documented in the Note; and

WHEREAS, Debtor is in default of its obligations pursuant to the Note; and

WHEREAS, as of December 31, 2011, the sum of Two Hundred Seven Thousand Ninety-Eight and no/100 Dollars (\$207,098.00) remains due and owing Secured Party from Debtor (the "Outstanding Debt"); and

WHEREAS, the Note provides Secured Party a security interest in all of Debtor's "patents and physical assets"; and

WHEREAS, Secured Party perfected his security interest via the filing of a Uniform Commercial Code Financing Statement with the Minnesota Secretary of State on or about February 10, 2012, Filing Number 201227190568; and

WHEREAS, Secured Party is currently the only secured creditor of Debtor; and

WHEREAS, Debtor is no longer a going business concern; and

WHEREAS, Debtor has no viable assets other than its rights as Licensee under that certain License Agreement dated July 30, 2008 (the "License Agreement"), a copy of which is attached hereto as Exhibit A, entered into by and between Debtor and Oxygenator Water Technologies, Inc. ("<u>OWT</u>"), a Minnesota corporation, pursuant to which Debtor holds certain perpetual, exclusive and royalty free licenses as further described in the License Agreement, any property of Debtor as set forth in the License Agreement, including but not limited to any Licensee Documents, Licensee Improvements, Licensee Patents, Licensee Products, Licensee Property or Licensee Technology, all as defined in the License Agreement and certain shares of

Agreement of Strict Foreclosure

**JA128** 

OWT's common stock held by Debtor (the "<u>OWT Stock</u>" or collectively with the License Agreement and the other property of Debtor set forth herein, the "<u>Collateral</u>"); and

WHEREAS, Secured Party, as Debtor's sole secured creditor, is entitled to foreclose on the Collateral securing the Note; and

WHEREAS, Secured Party has agreed and Debtor has consented to Secured Party's acceptance of the Collateral in full satisfaction of Debtor's obligations under the Note in accordance with Sections 9-620 through 9-622 of the Uniform Commercial Code (the "<u>UCC</u>"), as adopted in the State of Minnesota as Minnesota Statutes Sections 336.9-620 through 336.9-622.

#### AGREEMENTS

**NOW, THEREFORE,** in consideration of the foregoing, Debtor and Secured Party hereto agree as follows:

1. <u>Recitals</u>. Debtor hereby acknowledges that the recitals set forth above are true and correct and such recitals are hereby made a part of this Agreement.

2. <u>Conveyance of Collateral; Satisfaction of Outstanding Debt</u>. Pursuant to Minnesota Statutes Sections 336.9-620 through 336.9-622, Debtor assigns all right, title and interest in and to the Collateral to Secured Party, agrees to immediately surrender the Collateral to Secured Party and Secured Party shall retain the Collateral in full satisfaction of the Outstanding Debt. Debtor shall deliver the Stock Powers Certificate (Assignment Separate from Certificate) for the OWT Stock attached hereto as <u>Exhibit B</u> to Secured Party contemporaneous with Debtor's execution of this Agreement. Secured Party does <u>not</u> assume, and nothing herein shall be construed to obligate Secured Party to pay, any leases, liabilities or obligations of Debtor.

**3.** <u>Waiver</u>. Pursuant to Section 9-624 of the UCC, Minnesota Statutes Section 336.9-624, Debtor hereby waives and renounces all of its rights to notification under Section 9-611 of the UCC, Minnesota Statutes Section 336.9-611, or any other state in which any Collateral may be located as to the sale or other disposition by Secured Party of the Collateral and all rights under Sections 9-620 and 9-623 of the UCC, Minnesota Statutes Sections 336.9-620, regarding acceptance of collateral as discharge of the obligations of Debtor to Secured Party, mandatory disposition of the Collateral and the waiver of Debtor's rights to redeem collateral, respectively. Debtor further knowingly and intelligently waives any rights it may have to notice and a hearing before a court of competent jurisdiction.

#### 4. <u>Representations and Warranties</u>. Debtor represents and warrants the following:

4.1 Debtor has the power and is duly authorized to enter into and perform this Agreement, and Debtor has complied with and is in good standing with respect to all laws, statutes and ordinances of all federal, state and local governmental entities having jurisdiction over them. Debtor hereby represents and warrants that this Agreement is a legal, valid and binding agreement, enforceable in accordance

Agreement of Strict Foreclosure

**JA129** 

with its terms and shall be binding upon Debtor and its respective representatives, successors and assigns.

4.2 Debtor represents, warrants and covenants that it has valid title to all of the Collateral being turned over herewith.

5. [Intentionally Omitted].

6. <u>Representation by Counsel</u>. Debtor acknowledges that it has been represented by legal counsel of its choice, Jamie R. Pierce of Hinshaw & Culbertson, LLP, in connection with the execution and delivery of this Agreement.

7. <u>Notices</u>. Any notice required or permitted under this Agreement shall be in writing and shall be deemed to be given when and if sent by certified mail, return receipt requested, postage pre-paid, properly addressed as follows, or such other address as may hereafter be designated in writing by either of the parties:

Secured Party:

Debtor:

Roy Lecy c/o Nathan M. Brandenburg Siegel Brill, P.A. 100 Washington Avenue South, Suite 1300 Minneapolis, MN 55401

Aqua Innovations Incorporated 6101 Baker Road, Suite 206 Minnetonka, MN 55345

With a copy to:

Jamie R. Pierce Hinshaw & Culbertson, LLP 333 South Seventh Street, Suite 2000 Minneapolis, MN 55402

8. <u>Amendments, Waivers, Assignment</u>. No amendment, waiver or assignment of the provisions of this Agreement shall be effective unless the same shall be in writing and be signed by the party against whom it is to be enforced, and then such amendment, waiver or assignment shall be effective only in the specific interest and for the specific purpose which given.

9. <u>Necessary Documents</u>. The parties agree that they shall execute any and all documents necessary to carry out the terms and conditions of this Agreement.

10. <u>Governing Law; Venue</u>. This Agreement shall be governed and construed under and in accordance with the laws of the State of Minnesota. Any dispute arising under this Agreement

Agreement of Strict Foreclosure

3

JA130

and/or between the parties shall be venued in the state and federal courts located in Minneapolis, Minnesota.

11. <u>Entire Agreement</u>. This Agreement contains the entire Agreement by and between the parties hereto with respect to the transactions contemplated herein, which shall supersede any prior oral negotiations and agreements and shall be binding upon the parties hereto and their successors and assigns.

12. <u>Merger</u>. All prior oral and written communications, commitments, alleged commitments, promises, alleged promises, agreements and alleged agreements by or between Secured Party and Debtor are hereby merged into this Agreement.

13. <u>Severability</u>. If any part of this Agreement is held to be illegal, invalid or unenforceable, the remainder of this Agreement shall continue in full force and effect, notwithstanding such illegality, invalidity or unenforceability.

14. <u>Headings</u>. The section headings in this Agreement are included herein for convenience or reference only and shall not constitute a part of this Agreement for any other purposes.

15. <u>Successors and Assigns</u>. This Agreement shall be binding upon and shall inure to the benefit of the parties hereto and their respective successors and assigns, except that Debtor may not assign or transfer its rights or obligations hereunder without the prior written consent of Secured Party.

16. <u>Counterparts</u>. This Agreement may be executed in one or more counterparts, each of which shall be deemed to be an original and all of which shall constitute one and the same instrument.

17. <u>Indemnification</u>. Debtor agrees to indemnify, defend and hold Secured Party, his respective employees, agents, representatives and attorneys harmless from any claim or cause of action (except for a claim of fraud against Secured Party) by any third party based in whole or in part upon the terms of this Agreement or their actions or omissions in fulfilling or enforcing this Agreement or based upon any failure by Debtor to pay all taxes, or other indebtedness or fulfill any obligations they may have with other third parties which may be affected by this Agreement or the omissions or actions of the parties relative thereto.

[This space intentionally left blank; signature page follows.]

Agreement of Strict Foreclosure

**JA131** 

IN WITNESS WHEREOF, Secured Party and Debtor hereto have executed this Agreement as of the Effective Date.

Debtor:

**Aqua Innovations Incorporated** 

By: Its: EXECUTIVS

STATE OF MINNESOTA ) ) ss. COUNTY OF HENNEPIN )

This. document was acknowledged before me on October <u>4</u>, 2012, by <u>Allel Jush</u>, as <u>hig Cheutive Office</u> of Aqua Innovations Incorporated, a Minnesota corporation.

Notary Stamp:



Notary Signature  $\checkmark$ 

Roy H. Lecy

STATE OF MINNESOTA ) ) ss. COUNTY OF HENNEPIN )

This document was acknowledged before me on September \_\_\_\_\_, 2012, by Roy H. Lecy, an individual resident of the State of Minnesota.

Notary Stamp:

Notary Signature

M:\26141-001\Strict Foreclosure Agreement 05

Agreement of Strict Foreelosure

5

**JA132** 

IN WITNESS WHEREOF, Secured Party and Debtor hereto have executed this Agreement as of the Effective Date.

**Aqua Innovations Incorporated** Debtor: STATE OF MINNESOTA SS. ) COUNTY OF HENNEPIN ) This document was acknowledged before me on September \_\_\_\_, 2012, by \_\_\_\_\_, as \_\_\_\_\_\_ of Aqua Innovations Incorporated, a Minnesota corporation.

Notary Stamp:

Secured Party:

RAE	eag
Roy H. Lecy	8

STATE OF MINNESOTA COUNTY OF HENNEPIN )

SS.

Cotober This document was acknowledged before me on September <u>8</u>, 2012, by Roy H. Lecy, an individual resident of the State of Minnesota.

Notary Signature

Notary Stamp:



M:\26141-001\Strict Foreclosure Agreement 05

Agreement of Strict Foreclosure

Jau K. Haunt Notary Signature

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

Page 135

**JA133** 

5

CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 136 of 1320

# <u>EXHIBIT A</u> LICENSE AGREEMENT

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA134** 

CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 137 of 1320

# EXHIBIT A LICENSE AGREEMENT

# AMENDMENT TO LICENSE AGREEMENT

THIS AMENDMENT TO LICENSE AGREEMENT (the "Amendment"), is made, entered into and effective as of October 4, 2012, by and between OXYGENATOR WATER TECHNOLOGIES, INC. ("Licensor"), a Minnesota corporation doing business as Water D.O.G. Works, and ROY H. LECY ("Lecy"), and amends certain terms of that certain License Agreement dated July 30, 2008 (the "License Agreement"), entered into by and between Licensor and Aqua Innovations Incorporated ("Aqua"), a Minnesota corporation. Licensor and Lecy may be individually referred to herein as a "party" or collectively as the "parties."

WHEREAS, Licensor, as Licensor, and Aqua, as Licensee, entered into the License Agreement on or about July 30, 2008, a copy of which is attached hereto as <u>Exhibit A</u>; and

WHEREAS, pursuant to an Agreement of Strict Foreclosure dated October 4, 2012, Aqua conveyed all right, title and interest it held in the License Agreement to Lecy; and

WHEREAS, the parties desire to formally amend the License Agreement to reflect Lecy's interest in the Agreement via this Amendment,

# NOW THEREFORE, it is hereby agreed as follows:

1. <u>Incorporation of Recitals</u>. The recitals set forth above are true and correct and incorporated as if fully stated herein.

2. <u>Amendment of License Agreement</u>. All references in the License Agreement to Licensee shall mean Roy H. Lecy and not Aqua. Licensor hereby acknowledges the acquisition by Lecy of all right, title and interest of Aqua in the License Agreement and further acknowledges and agrees that the License Agreement is a binding contract in full force and effect and that Lecy may assign his interest therein at will.

3. <u>Remaining Terms in Full Force and Effect</u>. No other terms of the License Agreement or any schedule or exhibit thereto shall be amended or modified in any way and the License Agreement shall remain in full force and effect as amended via this Amendment and the parties hereby reaffirm their respective obligations thereunder.

Oxygenator Water Technologies, Inc.

By: 10-84-2012

Roy H, Lee

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA136

# PATENT ASSIGNMENT

# Electronic Version v1.1 Stylesheet Version v1.1

SUBMISSION TYPE: NEW ASSIGNMENT					
NATURE OF CONVEYANCE:		LICENSE			
CONVEYING PARTY DATA					
Name Execution Date				ן ו	
			10/04/2012	j	
RECEIVING PARTY	DATA				
Name:	O2 Marine Techn	ologi <b>es</b> , Inc.			
Street Address:	6651 Hazeltine B	oulevard			
City:	Excelsior				]
State/Country:	MINNESOTA				]
Postal Code:	55331				]
Property Type		Number			
<b></b>					ח
Patent Number: 6689					
Patent Number: 7396441					
Patent Number: 7670495					
CORRESPONDENCE DATA					
Fax Number:	6123396591 be sent via US Mai	when the fax attempt	is unsuccessful		
Phone:	6123376100		io unouccoolui.		
Email: nathanbrandenburg@siegelbrill.com					
Correspondent Name: Nathan M. Brandenburg					
Address Line 1: 100 Washington Avenue South					
Address Line 2: Suite 1300					
Address Line 4:	Address Line 4: Minneapolis, MINNESOTA 55401				
ATTORNEY DOCKE	T NUMBER:	25991-002			
NAME OF SUBMITTER: Nathan M. Brandenburg					
Signature: /nathanmbrandenburg/					

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 140 of 1320

Date:	04/30/2013
	This document serves as an Oath/Declaration (37 CFR 1.63).
Total Attachments: 5 source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page	ə2.tif ə3.tif ə4.tif

# PATENT SUBLICENSE AGREEMENT

THIS PATENT SUBLICENSE AGREEMENT (this "<u>Agreement</u>") is made, entered into and effective as of October 4, 2012 (the "<u>Effective Date</u>") by and between ROY H. LECY ("<u>Sublicensor</u>"), a Minnesota resident, and O2 MARINE TECHNOLOGIES, INC. ("<u>Sublicensee</u>"), a Minnesota corporation. Sublicensor or Sublicensee may be individually referred to herein as a "<u>party</u>" or collectively as the "<u>parties</u>."

WHEREAS, Sublicensor is the holder of certain patent licenses as set forth in a certain License Agreement dated July 30, 2008, entered into by and between Oxygenator Water Technologies, Inc. ("<u>QWT</u>"), as Licensor, and Aqua Innovations Incorporated ("<u>Aqua</u>"), as Licensee (the "<u>License Agreement</u>"), as amended via a certain Amendment to License Agreement dated October 4, 2012 (the "<u>Amendment</u>" or collectively with the License Agreement, the "<u>License Agreement</u>"), pursuant to which Roy H. Lecy acquired all right title and interest of Aqua in the License Agreement; and

WHEREAS, copies of the License Agreement and Amendment are attached hereto as Exhibit A.

NOW THEREFORE, the parties hereby agree as follows:

#### ARTICLE ONE GRANT OF SUBLICENSE

1.1 License. Sublicensor warrants he holds a valid license pursuant to the License Agreement for United States Patent Number 6,689,262 B2 issued on February 10, 2004, entitled "Microbubbles of oxygen," United States Patent Number 7,396,441 B2 issued on July 8, 2008, entitled "Flow-through oxygenator," and United States Patent Number 7,670,495 issued on March 2, 2010, entitled "Flow-through oxygenator" (collectively, the "Licensed Patents" or each a "Licensed Patent").

**1.2** Grant of Sublicense. Effective upon execution of this Agreement, and for consideration stated in Article Three of this Agreement, Sublicensor grants to Sublicensee a royalty based, limited, exclusive sublicense to make, sell, and offer for sale products covered by the Licensed Patents in the United States and to sell and offer for sale in any country products covered by the Licensed Patents (the "Licensed Products"), subject to the limitations set forth in this Agreement. The Licensed Patents are sublicensed "As Is" and without warranty of any kind by Sublicensor.

## ARTICLE TWO LICENSED FIELD

2.1 <u>Licensed Field</u> Sublicensee may sell and offer for sale only Licensed Products manufactured for retail sale and use in the sport fishing industry (the "<u>Licensed Field</u>") and for no other purposes. Sublicensee hereby agrees to offer for sale and to sell only the Licensed Products in the Licensed Field. Sublicensee acknowledges and agrees that if Sublicensee offers for sale or sells the Licensed Products outside the Licensed Field such activity by Sublicensee will be deemed a material breach of this Agreement and Sublicensor shall have the right to terminate this Agreement without notice.

1

# ARTICLE THREE ROYALTY

**3.1** <u>Royalty</u>. Sublicensee shall pay royalties to Sublicensor at a rate of five percent (5%) of the net receipts realized by Sublicensee upon any Licensed Products sold by Sublicensee. For purposes of this Agreement, "net receipts" shall mean gross sale proceeds, less cost of goods sold, freight, discounts offered by Sublicensee, returns and other costs or expenses incurred by Sublicensee in the manufacture and sale of the Licensed Products. Any royalties shall be paid to Sublicensor on a calendar quarterly basis, sixty (60) days after the end of each calendar quarter (May 30, August 30, December 30 and March 1 or 2).

Accounting. For all Licensed Products sold by Sublicensee, Sublicensee will account to 3.2 Sublicensor on a calendar quarterly basis, indicating the number of Licensed Products sold within thirty (30) days following the end of each calendar quarter for the term hereof. Sublicensee shall make such accountings to Sublicensor via paper or electronic accounting statements in a mutually accepted compatible format. Sublicensee agrees to keep and maintain true and accurate records and books of account in connection with all sales related to any such products and all transactions related thereto or otherwise contemplated under this Agreement, and shall retain all such records and books for a period of not less than five (5) years after each accounting to Sublicensor. Sublicensor, by its designated representative, shall have the right, upon reasonable written notice, and during normal office hours, to examine the books and records of Sublicensee, as the same pertain to the subject matter of this Agreement, and to make copies and extracts thereof. Sublicensee shall cooperate with Sublicensor's representatives to assist them in understanding all such material. If, as a result of any audit, it is determined that Sublicensee has understated the royalties due to Sublicensor by ten percent (10%) or more, Sublicensee shall pay to Sublicensor the amount by which royalties have been understated plus a ten percent (10%) penalty fee, and shall reimburse Sublicensor for the cost of the audit.

# ARTICLE FOUR PACKAGING

4.1 <u>Packaging</u>. Sublicensec shall use its own tradename or trade or servicemark on the packaging for the Licensed Products. In no event shall Sublicensee use any of Sublicensor's trademarks on the packaging for the Licensed Products.

## ARTICLE FIVE LICENSED PRODUCT REVIEW

5.1 Licensed Product Review. Sublicensor shall have the right to review the Licensed Products and packaging for the Licensed Products to be sold or offered for sale by Sublicensee prior to first sale of the Licensed Products and thereafter on a quarterly basis. Sublicensor shall have the right to provide suggestions concerning the quality and design of the Licensed Products, including packaging, to be sold or offered for sale by Sublicensee. If Sublicensor determines the quality of Licensed Products and/or packaging on the Licensed Products is unacceptable to Sublicensor, Sublicensor shall work with Sublicensee and offer suggestions to make the Licensed Products suitable and ready for market. Sublicensor and Sublicensee shall be willing to mutually work together in a reasonable manner without undue restriction.

2

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA140** 

# ARTICLE SIX MARKETING

6.1 <u>Marketing</u>. Sublicensee shall mark all Licensed Products made, used or sold under the terms of this Agreement with the following: "Protected by U.S. Patent Nos. 6,689,262, 7,396,441 and 7,670,495. Other Patents Pending."

# ARTICLE SEVEN TERM

7.1 <u>Term</u>. The sublicense so granted pursuant to this Agreement shall be effective from the Effective Date and shall terminate with respect to a Licensed Patent on the expiration of the Licensed Patent, subject to Section 7.2 of this Agreement.

7.2 <u>Termination</u>. Notwithstanding Section 7.1 of this Agreement, this Agreement shall terminate:

- 7.2.1 Upon the mutual agreement of the parties;
- 7.2.2 Upon ten (10) days' written notice from Sublicensor to Sublicensee upon Sublicensee's failure to pay any royalty due and owing Sublicensor;
- 7.2.3 Immediately if Sublicensee files of a petition of bankruptcy, or a petition or answer seeking reorganization, readjustment or rearrangement of its business or affairs under any law or governmental regulation relating to bankruptcy or insolvency;
- 7.2.4 Immediately if in Sublicensor's reasonable business judgment Sublicensee undertakes any action that derogates, disparages or impairs any of the Licensed Patents;
- 7.2.5 Immediately upon the terms of Section 2.1 of this Agreement; or
- 7.2.6 Upon ninety (90) days' written notice from Sublicensor to Sublicensee if Sublicensee fails to actively sell any Licensed Products or otherwise fails to actively and diligently utilize the sublicense granted by this Agreement.

# <u>ARTICLE EIGHT</u> INFRINGEMENT

**8.1** <u>Notice</u>. Sublicensee shall inform Sublicensor within thirty (30) days and in writing of any alleged infringement of the Licensed Patents by a third party.

8.2 <u>Legal Action: Mutual Cooperation</u>. Sublicensor shall have the right, but shall not be obligated, to prosecute at its own expense any infringement of the Licensed Patents and, in furtherance of such right, Sublicensee hereby agrees that Sublicensor may, if required by law or otherwise, include Sublicensee as a party plaintiff in any such suit, without expense to

3

**JA141** 

Sublicensee. The total cost of any such infringement action commenced or defended solely by Sublicensor shall be borne by Sublicensor, and Sublicensor shall keep any recovery or damages. In the event that any action alleging invalidity, non-enforceability, or non-infringement of any of the Licensed Patents shall be brought by a third party, Sublicensor, at its option, shall have the right to defend such actions.

# ARTICLE NINE INDEMNIFICATION

9.1 <u>Indemnification</u>. Sublicensee shall indemnify, defend, and hold harmless Sublicensor from any and all actions, claims, suits, losses, liabilities, damages, costs, fees, and expenses (including attorney fees) resulting from or arising out of the exercise of the Sublicensee's rights granted under this Agreement. This indemnification shall include, but is not limited to, any and all actions alleging product liability, patent infringement, or other type of intellectual property matter.

# ARTICLE TEN GENERAL TERMS

10.1 <u>Assignment</u>. The rights and sublicenses granted by Sublicensor in this Agreement are personal to Sublicensee and may not be assigned or otherwise transferred without the written consent of Sublicensor. Sublicensor may provide such consent upon request from Sublicensee for any assignment to a third party who is acquiring substantially all of the business assets of Sublicensee, but Sublicensor reserves the right to deny consent if the third party is a competitor of Sublicensor.

**10.2** <u>Sublicense</u>. The rights and license granted by Sublicensor in this Agreement may not be sublicensed by Sublicensee without the written consent of Sublicensor.

10.3 <u>Confidentiality</u>. Sublicensor and Sublicensee both agree the terms of this Agreement are confidential and shall not be disclosed to any third party.

10.4 <u>Governing Law</u>. This Agreement shall be construed and enforced according to the laws of the State of Minnesota. Any disputes arising out of, under, or relating to the negotiation, drafting, execution, validity, interpretation, breach, or enforcement of this Agreement shall be venued in the state or federal courts located in Minneapolis, Minnesota.

10.5 <u>Entire Agreement</u>. Sublicensee and Sublicensor acknowledge receipt of this Agreement and agree that with respect to the subject matter hereof this Agreement is the entire agreement of the parties and supersedes any previous oral or written communications or understandings, and that each provision has been given due consideration and accepted without duress.

10.6 <u>Attorneys' Fees</u>. In the event that any proceeding, suit or action is brought by any party under this Agreement to enforce any of its terms, it is agreed that the prevailing party shall be entitled to reasonable attorneys' fees to be fixed by the trial and appellate courts in any such proceeding or as incurred in the collection of any judgment.

4

**JA142** 

Counterparts. This Agreement may be signed in counterparts by the parties hereto with 10.7 the same force and effect as if the above parties signed the same original agreement. Facsimile and electronic copies and photocopies of the parties' signatures to this Agreement shall be valid and enforceable to the same extent as original signatures and the parties hereby waive any requirement that original signatures be produced as a condition of proving the validity of or otherwise enforcing this Agreement

IN WITNESS WHEREOF, Sublicensor and Sublicensee have executed this Agreement as of the Effective Date.

Sublicensor:

Roy H. Lecy

02 MARINE TECHNOLOGIES, INC.

By Dennis Clark

Its: President

Sublicensee:

5

**JA143** 

# PATENT ASSIGNMENT

# Electronic Version v1.1 Stylesheet Version v1.1

SUBMISSION TYPE:		NEW ASSIGNMENT		
NATURE OF CONVEYANCE:		ASSIGNMENT		
CONVEYING PARTY	Ó DATA			
		Name	Execution Date	
Aqua Innovations, In-	c.		10/04/2012	
RECEIVING PARTY	DATA			
Name:	Roy H Lecy			
Street Address:	2640 North Saund	ers Lake Drive		
City:	Minnetrista			
State/Country:	MINNESOTA			
Postal Code:	55364			
Property Type Patent Number: 6689		Num 262	iber	
PROPERTY NUMBE	RS Total: 3			
Patent Number: 6689		262		
Patent Number: 73964		441		
Patent Number: 7670495				
CORRESPONDENCE DATA				
Fax Number:	6123396591			
Correspondence will Phone:	612-337-610	when the fax attempt is unsucces	stul.	
Email:		enburg@siegelbrill.com		
Correspondent Name				
Address Line 1:		ton Avenue South		
Address Line 2: Suite 1300				
Address Line 4: Minneapolis, MINNESOTA 55446				
ATTORNEY DOCKET NUMBER:		26141-001		
NAME OF SUBMITTER:		Nathan M. Brandenburg		
Signature:		/nathanmbrandenburg/		

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 147 of 1320

Date:	04/30/2013				
	This document serves as an Oath/Declaration (37 CFR 1.63).				
Total Attachments: 27	Total Attachments: 27				
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source=OWT - Aqua License#page3.tif					
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# AMENDMENT TO LICENSE AGREEMENT

THIS AMENDMENT TO LICENSE AGREEMENT (the "<u>Amendment</u>"), is made, entered into and effective as of October 4, 2012, by and between OXYGENATOR WATER TECHNOLOGIES, INC. ("<u>Licensor</u>"), a Minnesota corporation doing business as Water D.O.G. Works, and ROY H. LECY ("<u>Lecy</u>"), and amends certain terms of that certain License Agreement dated July 30, 2008 (the "<u>License Agreement</u>"), entered into by and between Licensor and Aqua Innovations Incorporated ("<u>Aqua</u>"), a Minnesota corporation. Licensor and Lecy may be individually referred to herein as a "party" or collectively as the "<u>parties</u>."

WHEREAS, Licensor, as Licensor, and Aqua, as Licensee, entered into the License Agreement on or about July 30, 2008, a copy of which is attached hereto as <u>Exhibit A</u>; and

WHEREAS, pursuant to an Agreement of Strict Foreclosure dated October 4, 2012, Aqua conveyed all right, title and interest it held in the License Agreement to Lecy; and

WHEREAS, the parties desire to formally amend the License Agreement to reflect Lecy's interest in the Agreement via this Amendment.

# NOW THEREFORE, it is hereby agreed as follows:

1. <u>Incorporation of Recitals</u>. The recitals set forth above are true and correct and incorporated as if fully stated herein.

2. <u>Amendment of License Agreement</u>. All references in the License Agreement to Licensee shall mean Roy H. Lecy and not Aqua. Licensor hereby acknowledges the acquisition by Lecy of all right, title and interest of Aqua in the License Agreement and further acknowledges and agrees that the License Agreement is a binding contract in full force and effect and that Lecy may assign his interest therein at will.

3. <u>Remaining Terms in Full Force and Effect</u>. No other terms of the License Agreement or any schedule or exhibit thereto shall be amended or modified in any way and the License Agreement shall remain in full force and effect as amended via this Amendment and the parties hereby reaffirm their respective obligations thereunder.

Oxygenator Water Technologies, Inc.

Roy H, Lee By: 10-84-2012

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

# License Agreement

THIS AGREEMENT ("<u>Agreement</u>") is entered into this 30th day of July, 2008 (the "<u>Effective Date</u>"), by and between Oxygenator Water Technologies, Inc., a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensor</u>") and Aqua Innovations, Inc. a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensee</u>", and Licensor and Licensee each a "<u>Party</u>" and together the "<u>Parties</u>"). Initially capitalized terms defined in this Agreement shall have the meaning ascribed to them respectively herein.

#### WITNESSETH:

LICENSOR owns the technology for which patents have been issued and are pending with respect to electrolytic hydrolysis of water to increase its dissolved oxygen content. A more complete description of said technology, together with a description of the patents issued and currently pending for said technology, is set forth in Article 1 below and in Exhibit "A" attached hereto.

LICENSOR anticipates and intends that it will make additional discoveries and improvements to said technology, some of which may be patentable.

It is further anticipated by the parties that LICENSOR may make improvements to said technology and additional discoveries concerning other applications for said technology.

The parties desire that LICENSOR grant a perpetual, exclusive license to LICENSEE to develop and sell throughout the world certain products utilizing the technology LICENSOR has developed and may in the future develop, all according to the terms and conditions set forth in this Agreement.

The parties further desire that LICENSOR will retain the complete and entire right to develop and sell throughout the world in markets not licensed to LICENSEE hereunder products utilizing the technology LICENSOR has developed and may in the future develop or the technology that LICENSEE may develop in the future, also according to the terms and conditions set forth in this Agreement.

Thus, the parties have agreed to enter into a licensing arrangement by which each party will be entitled to benefit from the other party's patents, technology and know-how concerning electrolytic hydrolysis of water in the sale of products in certain markets.

**NOW, THEREFORE,** based on the foregoing and the mutual covenants and agreements herein contained, the parties hereby covenant and agree as follows:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

38 3<sup>5</sup> 10 3 EXHIBIT "B"

# **LICENSEE Markets**

All worldwide markets for:

- Waste Water Treatment
- Medical Applications
- Sport Fishing
- Aqua Culture
- Horticulture (consumer and commercial)
- Hydroponics

Markets excluded from license agreement (including but not limited to):

- Water Treatment (all applications except waste water)
- Fermentation
- Desalination
- Human Nutrition
- Animal Nutrition

# ARTICLE 1 DEFINITIONS

When used in this Agreement, the following terms have the meanings set forth below unless a different and common meaning of the term is clearly indicated by the context, and variants and derivatives of the following terms shall have correlative meanings:

"Agreement" has the meaning set forth in the preamble.

"LICENSOR Documents" has the meaning set forth in Section 2.6.

"LICENSOR Improvements" means all developments LICENSOR may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable, and which are invented, developed, discovered or otherwise acquired by LICENSOR and which LICENSOR may lawfully communicate to LICENSEE.

"*LICENSOR Markets*" means all uses for the LICENSOR Technology and the LICENSEE Technology other than in the LICENSEE Markets.

"LICENSOR Patents" means all of LICENSOR's patents (whether issued to LICENSOR or controlled by license rights or otherwise and whether such rights are held alone or jointly with others, and patents pending now, or during the term of this Agreement, issued to LICENSOR (by any country) relating to the LICENSOR Technology, including, but not limited to, those patents and those patents pending described on Exhibit A and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSOR Products*" means any product manufactured and/or sold or distributed by LICENSOR or a sub licensee of LICENSOR under any claim contained in the LICENSEE Patents.

"LICENSOR Property" means LICENSOR Patents, LICENSOR Improvements and LICENSOR Technology.

"LICENSOR Technology" means LICENSOR's unpatented technology and information now existing and relating to, and embodying LICENSOR's experience in electrolytic hydrolysis of water. LICENSOR Technology shall include the technical information in all current and future manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSOR to LICENSEE during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSOR imparting the same directly or giving LICENSEE access to any of LICENSOR's production facilities.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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"Effective Date" has the meaning set forth in the preamble.

"LICENSEE Documents" has the meaning set forth in Section 2.7.

"LICENSEE Improvements" means all developments LICENSEE may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable and which are invented, developed, discovered or otherwise acquired by LICENSEE and which LICENSEE may lawfully communicate to LICENSOR.

"*LICENSEE Markets*" means those markets for Licensee Products as are described in Exhibit B attached hereto.

"LICENSEE Patents" means all of LICENSEE's patents (whether issued to LICENSEE or controlled by license rights or otherwise and whether such rights are held alone or jointly with others) which may after the effective date of this Agreement be issued (by any country) relating to electrolytic hydrolysis of water and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSEE Products*" means any product manufactured and/or sold or distributed to any party other than LICENSOR by LICENSEE or a sublicense of LICENSEE in conformity with the terms of this Agreement, including, but not limited to, any product which is based on any claim or thing contained in any LICENSOR Property.

"LICENSEE Property" means LICENSEE Patents, LICENSEE Improvements and LICENSEE Technology.

"LICENSEE Technology" means LICENSEE's unpatented technology and information which LICENSEE may develop relating to, and embodying LICENSEE's experience in, the manufacturing, the processing, quality control, and sale of the LICENSEE Products. LICENSEE Technology shall include the technical information in all manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSEE to LICENSOR during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSEE imparting the same directly or giving LICENSOR access to any of LICENSEE's production facilities.

"Territory" means the world,

# ARTICLE 2 MARKETS AND LICENSING

2.1. Exclusive Markets. The parties agree that unless properly terminated by LICENSOR pursuant to Section 5.1 below, LICENSEE will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSEE Markets in the Territory. The parties further agree that LICENSOR will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSOR Markets in the Territory. LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSOR Products in the LICENSOR Markets in the Territory. LICENSEE may not, directly or indirectly, distribute in any manner any product which competes with the LICENSEE Products in any manner nor may LICENSEE assist or have any interest in any third party distributing any such products through licensing or assignment of technology to any such third party or by any other means.

2.2. Exclusive License to LICENSEE. Subject to the terms and conditions of this Agreement, LICENSOR hereby confers upon LICENSEE the sole and exclusive license, with the right of sublicense, under the LICENSOR Property, to make, have made, use and sell the LICENSEE Products in the LICENSEE Markets in the Territory and to prevent infringement of the LICENSOR Patents, and to prevent unauthorized use and disclosure of the LICENSOR Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSEE Products.

2.3. Exclusive License to LICENSOR. Subject to the terms and conditions of this Agreement, LICENSEE hereby confers upon LICENSOR the sole and exclusive, royalty-free license, with the right of sublicense, under the LICENSEE Property, to make, have made, use and sell the LICENSOR Products in the LICENSOR Markets in the Territory and to prevent infringement of the LICENSEE Patents, and to prevent unauthorized use and disclosure of the LICENSEE Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSOR Products.

**2.4. Product Markings.** The Parties shall insure that all LICENSOR Products and all LICENSEE Products are marked with any applicable patent number and all labeling and other product information shall be marked in such manner as to conform with the patent laws and practices of the country of sale.

2.5 Transfer of Technology by LICENSOR. As promptly as practicable after the execution of this Agreement, LICENSOR shall deliver to LICENSEE all information concerning the LICENSOR Property. LICENSOR also promptly shall deliver to LICENSEE all future information it acquires concerning the LICENSOR Property. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSOR, shall be referred to as "LICENSOR Documents". LICENSEE shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSOR Documents in such manner as to maximize the value of the LICENSOR Patents, the LICENSOR Technology and the LICENSOR Improvements; without limiting the generality of the foregoing, LICENSEE shall, and shall cause its employees and

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA151

representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSOR Documents. Upon 10 days advance notice and at reasonable times, LICENSOR shall permit LICENSEE access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSOR to answer fully such questions as LICENSEE may have with a view to transferring the LICENSOR Property. Nothing in this Section 2.6 shall require LICENSOR to disclose to LICENSEE any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

#### 2.6. Transfer of Technology by LICENSEE. As promptly as practicable after

LICENSEE develops, discovers or otherwise comes into possession of LICENSEE Patents, LICENSEE Improvements and/or LICENSEE Technology, LICENSEE shall deliver to LICENSOR all information concerning same. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSEE, shall be referred to as "LICENSEE Documents." LICENSOR shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSEE Documents in such manner as to maximize the value of the LICENSEE Patents, the LICENSEE Technology and the LICENSEE Improvements; without limiting the generality of the foregoing, LICENSOR shall, and shall cause its employees and representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSEE Documents. Upon 10 days advance notice and at reasonable times, LICENSEE shall permit LICENSOR access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSEE to answer fully such questions as LICENSOR may have with a view to transferring to LICENSOR the LICENSEE Property. Nothing in this Section 2.7 shall require LICENSEE to disclose to LICENSOR any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

2.7. Further Prosecution of Patents. LICENSOR will continue with the prompt prosecution of all pending patent applications filed by LICENSOR as detailed on Schedule "A", so long as it is commercially reasonable to do so, and LICENSOR will periodically advise LICENSEE of the status of such prosecutions. As soon as practical, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSOR Patents. In the event that LICENSEE files an application for a patent(s) covering electrolytic hydrolysis of water, LICENSEE will periodically advise LICENSOR of the status of the prosecution of any such patent. As soon as practical after any such application by LICENSEE, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSEE Patents. From the date of this Agreement, all expenses incurred in filing for and maintaining protection in those countries mutually agreed upon (other than expenses of prosecuting the original patent application in the first jurisdiction, which will be the responsibility of the Party filing the patent application) will be shared equally by the Parties. Either Party may seek protection in any country not mutually agreed upon by paying the full amount of the cost thereof. A party seeking such additional protection will receive the full cooperation of the other Party (other than in paying the expenses thereof) in protecting all patents in any such other country.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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2.8. Additional Covenants. Each of LICENSOR and LICENSEE shall faithfully comply with their respective obligations under this Agreement and shall incorporate all terms and conditions required by this Agreement in any contracts with third parties to whom access to the LICENSOR Property or the LICENSEE Property, as the case may be, may (but only in accordance with this Agreement) be given. Each of LICENSOR and LICENSEE shall indemnify and hold harmless the other Party and its successors and assigns from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on such Party, and arising out of or in connection with or resulting from the marketing, sale or use of the imdemnifying Party's product(s), including any advertising or other promotional activities related thereto.

**2.9. Infringement Actions.** Neither LICENSOR nor LICENSEE will have any responsibility to the other Party for any damage or expense incurred by such other Party which arises from any action, claim or cause of action brought by any person as the result of any alleged patent infringement or trade secret misappropriation by reason of such other Party's manufacture, use or sale of any product under any of the licenses conferred hereby.

2.10. LICENSEE's Rights in Event of Third Party Infringement. LICENSEE shall have the right, in LICENSOR's name (if required by law, otherwise, in LICENSEE's name) but at LICENSEE's sole expense, to sue third parties in the LICENSEE Markets for infringements of the LICENSOR Patents and misappropriation of the LICENSOR Technology and unpatented LICENSOR Improvements, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. LICENSOR may, if it so elects, join in any such suit as a plaintiff. All damages, awards or settlement proceeds in such suit shall be LICENSEE's. If LICENSEE, after notice from LICENSOR of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSOR, in its own name (or, if required by law, in its and LICENSEE's name) and at its own expense, may sue therefore, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSOR shall promptly reimburse LICENSEE for said suitassociated direct expenses upon presentation of LICENSEE's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's.

2.11. LICENSOR's Rights in Event of Third Party Infringement. LICENSOR

shall have the right, in LICENSEE's name (if required by law, otherwise, in LICENSOR's name) but at LICENSOR's sole expense, to sue third parties in the LICENSOR Markets for infringements of the LICENSEE Patents and misappropriation of the LICENSEE Technology and unpatented LICENSEE Improvements, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSEE may, if it so elects, join in any such suit as a plaintiff. LICENSOR shall promptly reimburse LICENSEE for said suit-associated direct expenses upon presentation of LICENSEE's itemized

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA153

statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's. If LICENSOR, after notice from LICENSEE of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSEE, in its own name (or, if required by law, in its and LICENSOR's name) and at its own expense, may sue therefore, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSEE's.

**2.12. LICENSEE Royalty Payment.** None. License is granted without cost to LICENSEE.

# ARTICLE 3 INDEMNIFICATION

**3.1.** Indemnification by LICENSEE. LICENSEE shall indemnify and hold LICENSOR and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSOR arising out of or in connection with or resulting from the marketing, sale or use of the LICENSEE Products, including any advertising or other promotional activities related thereto. LICENSOR shall be an added insured party to LICENSEE's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSEE shall procure and have in place no later than the date on which LICENSEE first makes a delivery of any of the LICENSEE Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSOR is provided at least thirty (30) days advance written notice.

**3.2. Indemnification by LICENSOR.** LICENSOR shall indemnify and hold LICENSEE and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSEE arising out of or in connection with or resulting from the marketing, sale or use of the LICENSOR Products, including any advertising or other promotional activities related thereto. At such time, if any, as LICENSOR shall sell LICENSOR Products, LICENSOR shall add LICENSEE as an added insured party to LICENSOR's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSOR shall procure and have in place no later than the date on which LICENSOR first makes a delivery of any of the LICENSOR Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSEE is provided at least thirty (30) days advance written notice.

#### ARTICLE 4 CONFIDENTIALITY

4.1. Restrictions on Use and Disclosure of LICENSOR Property by LICENSEE. LICENSEE shall use the LICENSOR Property in confidence and shall not

disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate: (a) if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSEE, or (b) upon the expiration of the obligation of LICENSEE under this Agreement to pay royalties to LICENSOR.

4.2. Restrictions on Use and Disclosure of LICENSEE Property by LICENSOR. LICENSOR shall use the LICENSEE Property in confidence and shall not disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSOR.

#### 4.3. Employees; Third Parties Etc. In order to faithfully perform their

respective obligations under sections 4.1 and 4.2, the Parties shall limit access to the other Party's Property to only those of its officers, employees and agents who shall have a need to receive or have access to that portion, and then only for the purposes of the practice under the licenses conferred by this Agreement. Each Party will require any third party, to whom access may be authorized under this Agreement, to execute an appropriate confidentiality agreement.

4.4. Authorized Required Disclosures. Nothing in this Article 4 shall prevent a Party: (a) from complying (but only to the narrowest extent required by law and regulation and with due notice on any submissions to governmental agencies of the confidential or proprietary status of the information with a view toward restricting access to, and use or disclosure by, third parties) with reasonable requirements of governmental agencies to disclose information in order to receive legally required consents or permissions to manufacture or sell that Party's Products; or (b) from disclosing information under court order, but only after having made all reasonable efforts to secure the court's order to (i) limit production, use and disclosure of said information for the purposes of the case and to the narrowest class of disclosures practicable under the circumstances and (ii) hold all proceedings in camera with a sealed record.

## ARTICLE 5 RESOLUTION OF DISPUTES

All claims, disputes and other matters in question arising out of, or relating to, this Agreement or the performance thereof shall be submitted to, and determined by, arbitration if good faith negotiations between the parties do not resolve such claim, dispute or other matter within 60 days. Such arbitration shall proceed in accordance with the Commercial Arbitration Rules of the American Arbitration Association then pertaining (the "Rules"), insofar as such Rules are not inconsistent with the provisions expressly set forth in this Agreement, unless the parties mutually agree otherwise, and pursuant to the following procedures:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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(a) Notice of the demand for arbitration shall be filed in writing with the other Member and with the American Arbitration Association. Each Member shall appoint an arbitrator, and those party-appointed arbitrators shall appoint a third neutral arbitrator within 10 days. If the party-appointed arbitrators fail to appoint a third, neutral arbitrator within 10 days, such third, neutral arbitrator shall be appointed by the American Arbitration Association in accordance with the Rules. A determination by a majority of the panel shall be binding.

(b) Reasonable discovery shall be allowed in arbitration.

(c) All proceedings before the arbitrators shall be held in Minneapolis, Minnesota. The governing law shall be as specified in Section 8.1 below.

(d) The costs and fees of the arbitration, including attorneys' fees, shall be allocated by the arbitrators.

(e) The award rendered by the arbitrators shall be final and judgment may be entered in accordance with applicable law and in any court having jurisdiction thereof.

#### ARTICLE 6 NOTICES

**6.1.** Notices. All communications, demands, notices or objections required or permitted to be given or served under this Agreement shall be in writing and shall be deemed to have been duly given or made only if delivered in person, deposited in the United States mail, postage prepaid, for mailing by certified or registered mail, return receipt requested, or delivered by prepaid overnight courier service, addressed to the appropriate party as follows:

- If to LICENSOR: Richard Disrud, COO Aqua Innovations, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435
- If to LICENSEE: Jeffrey Brink, CEO Oxygenator Water Technology, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435

Either party may change its address by giving notice in writing, stating the new address, to the other Party as provided in the foregoing manner. Commencing on the tenth (10th) day after the giving of such notice, such newly designated address shall be such Party's address for the purpose of all communications, demands, notices or objections required or permitted to be given or served under this Agreement.

# ARTICLE 7 MISCELLANEOUS

**7.1. Governing Law; Court Proceedings.** The validity, performance, and all matters relating to the interpretation and effect of this Agreement shall be governed by the internal law in effect in the State of Minnesota without regard to principles of law (such as "conflicts of law") that might make the law of some other jurisdiction applicable. Without limiting the terms set forth in Article 6 with respect to the resolution of disputes, each Party agrees to the exclusive and irrevocable jurisdiction arising out of or in any way related to this Agreement which may be brought in a court of law and both parties agree that personal service from any such court may be effectively served upon a party at the respective addresses set forth in Section 7.1.

**7.2. Exhibits.** Exhibits, schedules and annexes referred to in this Agreement and attached hereto are incorporated herein in full by this reference as if each of such exhibits, schedules or annexes were set forth in the body of this Agreement and duly executed by the parties hereto.

**7.3. Additional Documents and Acts.** Each party agrees that it will use all reasonable efforts to take, or cause to be taken, all actions and to do, or cause to be done, all things necessary, proper or advisable, including, but not limited to, the execution of additional documents and instruments, to consummate, make effective and carry out the transactions contemplated by this Agreement.

7.4. Amendment, Modification or Waiver. No amendment, modification or waiver of any condition, provision or term of this Agreement shall be valid or of any effect unless made in writing, signed by the party or parties to be bound or its duly authorized representative and specifying with particularity the nature and extent of such amendment, modification or waiver. Any waiver by any party of a default of another party shall not affect or impair any right arising from any subsequent default.

**7.5. Severable Provisions.** Whenever possible, each provision of this Agreement will be interpreted in such manner as to be effective and valid under applicable law, but if any provision of this Agreement is held to be invalid, illegal or unenforceable under any applicable law or rule in any jurisdiction, such provision will be ineffective only to the extent of such invalidity, illegality, or unenforceability in such jurisdiction, without invalidating the remainder of this Agreement in such jurisdiction or any provision hereof in any other jurisdiction.

**7.6. Entire Agreement.** This Agreement contains the entire understanding of the parties hereto in respect of the transactions contemplated hereby and supersedes all prior agreements and understandings between the parties with respect to such subject matter.

7.7. Captions, Headings, Titles or References to Gender. All captions, headings

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

or titles in the paragraphs or sections of this Agreement are inserted for convenience of reference only and shall not constitute a part of this Agreement or as a limitation of the scope of the particular paragraphs or sections to which they apply. Where appropriate, the masculine gender may be read as the feminine gender or the neuter gender, the feminine gender may be read as the masculine gender or the neuter gender and the neuter gender may be read as the masculine gender or the feminine gender.

**7.8.** Counterparts. This Agreement may be executed in two (2) or more counterparts, each of which shall be considered one and the same Agreement and shall become effective when one or more counterparts have been signed by each of the parties and delivered to the other parties.

IN WITNESS WHEREOF, the parties have executed this Agreement on the date first written above.

AQUA INNOVATIONS, INC.

1.03 =

Dick Disrud its COO

**OXYGENATOR WATER TECHNOLOGIES, INC.** 

Jeff Brink its CEO

EXHIBIT "A"

# DESCRIPTION OF LICENSOR PATENTS AND PATENTS PENDING

## United States Patent Number: US 6,689,262 B2 Date of Patent: February 10, 2004 Name: Microbubbles of Oxygen Application Number: 10/372,017

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen.

United States Patent Number: US 7,396,441 B2 Publication Date: July 8, 2008 Name: Flow-Thru Oxygenator Application Number: 10/732,326

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen. A flow-through model for oxygenating flowing water. The use of supersaturated water for enhancing the growth of plants. Method of applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture. The treatment of waste water by raising the dissolved oxygen with the use of oxygen emitter.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 162 of 1320

#### United States Patent & Trademark Office

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<ul> <li>Resources &amp; Public Notices</li> </ul>	Examiner Name:	ALLEN, CAMERON J	Location: 👁	ELECTRONIC
Patent Searches	Group Art Unit:	1797	Location Date:	-
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<sup>1</sup> Copies, Products & Services	Attorney Docket Number:	4056.020503	Earliest Publication Date:	07-31-2008
Diher	Class / Subclass:	210/748	Patent Number:	7,670,495
opyrights rademarks slidy 6 Law soorts	First Named Inventor:	James Andrew Senkiw , Minneapolis, MN (US)	Issue Date of Patent:	03-02-2010
	Title of Invention:	DOW-THDOIC	+ OXYGENATOR	

Call the Patent Electronic Business Center at (866) 217-9197 (toll free) or e-mail EBC@uspto.gov for specific questions about Patent Application Information Retrieval (PAIR).
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THIS PATENT ISSUED 3/2/2010. In DISCUSSIONS WITH PAUL HAMN, IT GIVES US BREAD COVERAGE.

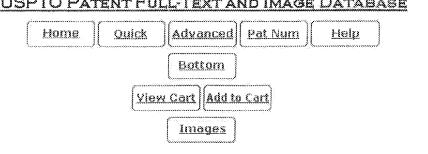
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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 163 of 1320

United States Patent: 7670495

Page 1 of 12



USPTO PATENT FULL-TEXT AND IMAGE DATABASE

**United States Patent** Senkiw

7,670,495 March 2, 2010

(1 of 1)

Flow-through oxygenator

#### Abstract

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flowthrough model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

Inventors: Senkiw; James Andrew (Minncapolis, MN) Assignce: Oxygenator Water Technologies, Inc. (Minnetonka, MN) Appl. No.: 12/023,431 Filed: January 31, 2008

#### **Related U.S. Patent Documents**

<b>Application</b> Number	<b>Filing Date</b>	Patent Number	<b>Issue Date</b>
10732326	Dec., 2003	7396441	
10372017	Feb., 2004	6689262	
60358534	Feb., 2002		

Current U.S. Class:	204/232; 204/245; 205/628; 210/243; 210/600
Current International Class:	C02F 1/48 (20060101); C02F 1/00 (20060101); C25B
	1/02 (20060101); C25B 1/04 (20060101)
Field of Search:	210/748,600,243 204/278,242,243,275.1,232,286.1,554,660
	205/633-638

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Page 2 of 12

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Mohyuddin Mirza et al., "Effect of Oxygenated Water on the Growth & Biomass Development of Seedless Cucumbers and Tomato Seedlings under Greenhouse Conditions," Seair Diffusion Systems, 2003, 5 pages, www.seair.ca. cited by other.

Primary Examiner: Griffin; Walter D Assistant Examiner: Allen; Cameron J Attorney, Agent or Firm: Patterson, Thuente, Skaar & Christensen, P.A.

#### Parent Case Text

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JA162

Page 3 of 12

RELATED APPLICATIONS

This application is a division of application Ser. No. 10/732,326 filed Dec. 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

Claims	
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The invention claimed is:

1. A method for treating waste water comprising; providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other, placing the emitter within a conduit; and passing waste water through the conduit.

2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breading the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.

5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.

7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.

8. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising: inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.

9. A method for lowering the biologic oxygen demand of polluted water comprising: passing the polluted water through a vessel containing the emitter of claim 2.

10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.

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#### Page 4 of 12

11. The emitter of claim 2, further comprising a timer control.

12. The emitter of claim 2, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.

Description

# FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

TABLE-US-00001 AT THE CATHODE: 4H.sub.2O + 4e.sup.- .fwdarw. 4OH.sup.- + 2H.sub.2 AT THE ANODE: 2H.sub.2O .fwdarw. O.sub.2 + 4H.sup.+ + 4e.sup.- NET REACTION: 6H.sub.2O .fwdarw. 4OH.sup.- + 4H.sup.+ ++ 2H.sub.2 + O.sub.2

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Page 5 of 12

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

# SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 6 of 12

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

# BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the O.sub.2 emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the O.sub.2 emitter.

FIG. 4 shows a funnel or pyramid variation of the O.sub.2 emitter.

FIG. 5 shows a multilayer sandwich O.sub.2 emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

# DETAILED DESCRIPTION OF THE INVENTION

Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O.sub.2 emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O.sub.2. In the special dimensions of the invention, as explained in more detail in the following examples, O.sub.2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H.sub.2 formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 8 of 12

shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon.RTM., polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an O.sub.2 emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### **EXAMPLE 2**

#### Measurement of O.sub.2 Bubbles

Attempts were made to measure the diameter of the O.sub.2 bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed O.sub.2 bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### EXAMPLE 3

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Page 9 of 12

Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H.sub.2) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

 TABLE-US-00002 TABLE I Emitter Model Gallons Volts Amps Max. Ave Watts Bait keeper 5 6 0.090
 0.060 0.36 Livewell 32 12 0.180 0.120 1.44 OEM 2 inch 10 12 0.210 0.120 1.44 Bait store 70 12 0.180

 0.180 2.16 Double cycle 2 12 0.180 0.180 2.16 OEM 3 inch 50 12 0.500 0.265 3.48 OEM 4 inch 80 12
 0.980 0.410 4.92 Water pail 2 24 1.200 1.200 28.80 Plate 250 12 5.000 2.500 30.00

**EXAMPLE 4** 

Multilayer Sandwich O.sub.2 Emitter

An O.sub.2 emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

TABLE-US-00003 cathode screen 0.045 inches thick nylon spacer 0.053 inches thick anode grid 0.035 inches thick nylon spacer 0.053 inches thick cathode screen 0.045 inches thick,

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

**EXAMPLE 5** 

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Page 10 of 12

Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in one-inch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 11 of 12

half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

TABLE-US-00004 TABLE II Control, grams Test, grams tomatoes from tomatoes from eight plants/ seven plants/ Week of: cumulative total cumulative total July 27 240 400 August 3 180 420 2910 3310 August 10 905 1325 1830 5140 August 17 410 1735 2590 7730 August 24 3300 5035 2470 10200 August 31 4150 9175 1580 11780 September 15 not weighed 3710 15490 Final Harvest 6435 15620 8895 24385 September 24

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

#### **EXAMPLE 6**

Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120.degree. angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2.degree. C. but the flowing water cooled slightly to 11 or 11.5.degree. C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE-US-00005 TABLE III ACTIVE DO OF\* ELECTRODE CURRENT, FLOW RATE SAMPLE AT MODEL AREA, SQ.IN. VOLTAGE AMPS. GAL/MINUTE ONE MINUTE 2-Inch "T" 2 28.3 0.72 12 N/A 3-inch "T" 3 28.3 1.75 12 N/A 2-plate Tube 20 28.3 9.1 12 8.4 3-Plate tube 30 28.3 12.8 12 9.6

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Page 12 of 12

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The oneminute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

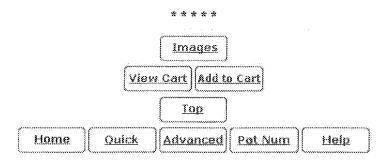
It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

EXAMPLE 7

Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.



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Date Mailed: 08/22/2007

# NOTICE OF ACCEPTANCE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 08/17/2007.

The Power of Attorney in this application is accepted. Correspondence in this application will be mailed to the above address as provided by 37 CFR 1.33.

Office of Initial Patent Examination (571) 272-4000, or 1-800-PTO-9199 OFFICE COPY

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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• •			Page 1 of 1
UNITED STATE	s Patent and Tradema	UNITED STA United States Address COMMI P.O. Box I	, Virginia 22313-1450
APPLICATION NUMBER	FILING OR 371 (c) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
10/372,017	02/21/2003	James Andrew Senkiw	AQI001US1
Kathleen R. Terry 1666 COFFMAN STREET SUITE 314		+OC0000000	CONFIRMATION NO. 9911

Date Mailed: 08/22/2007

/i

# NOTICE REGARDING CHANGE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 08/17/2007.

FALCON HEIGHTS, MN 55108

• The Power of Attorney to you in this application has been revoked by the assignee who has intervened as provided by 37 CFR 3.71. Future correspondence will be mailed to the new address of record(37 CFR 1.33).

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 177 of 1320

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	DATÉ:	August 17, 2007			
	TO:	Commissioner for Paten	ts	FAX #: 571-273-830	0
	Patent No.: Applicant: Due Date:	6,689,262 James Andrew S n/a	Senkiw	OUR REF.: 4056.04	US01
	FROM: PHONE #:	J. Paul Haun 612-349-3009			
	Attach	ned please find the following	ng for filing in the above-identi:	fied application:	
	1. 2.	Substitution of Attorney Certificate Under 37 CF		A A	
	·	CERTIFIC	J. Paul Haun Registration No. 53,00		
	I hereby certify th on the date show		d by facsimile to the U.S. Parent and T	addinant Office, Fax No. 1	571-273-8300
	Date	<u> </u>	J. Paul Haun	g	<b></b>
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# AUG 17 2007

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Attomey Docket No.: 4056.04US01

Confirmation No.: 9911

Application No.: 10/372,017

Group Art Unit: 1746

6,689,262 Patent No .:

Filed: December 10, 2003

MICROBUBBLES OF OXYGEN For:

James Andrew Senkiw

#### SUBSTITUTION OF ATTORNEY

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

I hereby appoint the practitioners associated with Customer Number 24113 to prosecute

this application and to transact all business in the Patent and Trademark Office connected therewith.

Address all telephone calls to: J. Paul Haun at telephone number (612) 349-3009.

Address all correspondence to:

Customer Number 24113 J. Paul Haun Patterson, Thuente, Skaar & Christensen, P.A. 4800 IDS Center, 80 South 8th Street Minneapolis, Minnesota 55402-2100

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

CERTIFICATE OF MAILING

I hereby certify that this document is being deposited with the United States Posmail in an envelope addressed to: Commissioner for Patents,  $R_{\rm IO}$ . Box 1450, Alex h sufficient postage as first class 13-1450 on ານໄ ໄດ້ແກ Date of De

PAGE 2/5\* RCVD AT 8/17/2007 3:21:40 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/22 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):01-40

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 179 of 1320

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Patent No. 6,689,262

SRMd

Please reference Attorney Docket No. 4056.04US01 on all correspondence. Additionally, please charge any future fees to Deposit Account No. 16-0631.

All previous powers of attorney granted in this case are hereby revoked.

Aqua Innovations, Inc., Assignee

8-15-07 Date:

Signature,

Name Printed or Typed

Κı

()(Title

PAGE 3/5\* RCVD AT 8/17/2007 3:21:40 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/22 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):01-40

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 180 of 1320

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	AUG 17 2007 PATENT
IN THE UNITED STATES PAT	ENT AND TRADEMARK OFFICE
In re the application of:	Attorney Docket No.: 4056.04US01
James Andrew Senkiw	Confirmation No.: 9911

Patent No.: 6,689,262

Filed: December 10, 2003

For: MICROBUBBLES OF OXYGEN

#### CERTIFICATE UNDER 37 CFR § 3.73(b)

Application No.: 10/372,017

Group Art Unit: 1746

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Ŷ

Aqua Innovations, Inc., a corporation, states that it is the assignee of the entire right, title and interest in the patent application identified above by virtue of either:

 A. [X] An assignment from the inventor(s) of the patent application identified above. The assignment was recorded in the Patent and Trademark Office at Reel 019690, Frame 0523, or for which a copy thereof is attached.

OR

 B. [] A chain of title from the inventor(s), of the patent application identified above, to the current assignee as shown below:

1. From \_\_\_\_\_ to \_\_\_\_

The document was recorded in the Patent and Trademark

Office at Reel \_\_\_\_\_, Frame \_\_\_\_\_ or for which a copy thereof is attached.

PAGE 4/5 \* RCVD AT 8/17/2007 3:21:40 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/22 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):01-40

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 181 of 1320

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Patent No. 6,689,262

2. From \_\_\_\_\_ to \_\_\_\_\_

The document was recorded in the Patent and Trademark Office at Reel \_\_\_\_\_, Frame \_\_\_\_\_ or for which a copy thereof is attached.

3. From \_\_\_\_\_ to \_\_\_\_\_

The document was recorded in the Patent and Trademark Office at Reel \_\_\_\_\_, Frame \_\_\_\_\_ or for which a copy thereof is attached.

[ ] Additional documents in the chain of title are listed on a supplemental sheet.

[ ] Copies of assignments or other documents in the chain of title are attached.

The undersigned (whose title is supplied below) is empowered to sign this statement on

behalf of the assignee.

8-15-07 Date:

Signature

Name Printed or Typed

Title

2

PAGE 5/5 \* RCVD AT 8/17/2007 3:21:40 PM [Eastern Daylight Time] \* SVR: USPTO-EFXRF-2/22 \* DNIS: 2738300 \* CSID: 6123499266 \* DURATION (mm-ss): 01-40

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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JUL 27 2006 H		nt and Trademark Office	se through 12/31/200 ; U.S. DEPARTMEN	T OF COMMERCE
CHANGE OF	Patent Number	6,689	262	
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Commissioner for Patents P.O. Box 1450	First Named Inventor	Fames	Andrew	Senkiw
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Assignee of record of the entire interest Statement under 37 CFR 3.73(b) is end	. See 37 CFR 3.71. losed. (Form PTO/SB/96)			
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if more than one signature is required, see below*.				
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This collection of information is required by 37 CFR 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application for not the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop Post Issue, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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10/372,017 TITLE OF INVENTION: N	02/21/2003 AICROBUBBLES OF OXYC	James Andr	ew Senkiw	AQIONIUSI	9911	
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			1746	204-278500
			DATE MAILED: 09/30/200	3
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
			AQ1001US1	

TITLE OF INVENTION: MICROBUBBLES OF OXYGEN

APPLN. TYPE	SMALL ENTITY	ISSUE FEE	PUBLICATION FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	nonprovisional YES		\$300	\$950	12/30/2003

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. <u>PROSECUTION ON THE MERITS IS CLOSED</u>. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

AN EQUIVALENT) MUST BE RETURNED WITHIN THIS PERIOD EVEN IF NO FEE IS DUE OR THE APPLICATION WILL BE REGARDED AS ABANDONED.

#### HOW TO REPLY TO THIS NOTICE:

I. Review the SMALL ENTITY status shown above.

If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status: A. If the status is the same, pay the TOTAL FEE(S) DUE shown above.	If the SMALL ENTITY is shown as NO: A. Pay TOTAL FEE(S) DUE shown above, or
B. If the status is changed, pay the PUBLICATION FEE (if required) and twice the amount of the ISSUE FEE shown above and notify the United States Patent and Trademark Office of the change in status, or	<ul> <li>B. If applicant claimed SMALL ENTITY status before, or is now claiming SMALL ENTITY status, check the box below and enclose the PUBLICATION FEE and 1/2 the ISSUE FEE shown above.</li> <li>Applicant claims SMALL ENTITY status. See 37 CFR 1.27.</li> </ul>

II. PART B - FEE(S) TRANSMITTAL should be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). Even if the fee(s) have already been paid, Part B - Fee(s) Transmittal should be completed and returned. If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

Page 1 of 4

PTOL-85 (Rev. 08/03) Approved for use through 04/30/2004.

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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Kathleen R. Terry 2417 Como Avenu			BELL, B	RUCE F
St. Paul, MN 55103	-		ART UNIT	PAPER NUMBER
			1746	
			DATE MAILED: 09/30/200	3

### Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (application filed on or after May 29, 2000)

The Patent Term Adjustment to date is 0 day(s). If the issue fee is paid on the date that is three months after the mailing date of this notice and the patent issues on the Tuesday before the date that is 28 weeks (six and a half months) after the mailing date of this notice, the Patent Term Adjustment will be 0 day(s).

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) system (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (703) 305-1383. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at (703) 305-8283.

Page 3 of 4

PTOL-85 (Rev. 08/03) Approved for use through 04/30/2004.

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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Kathleen R. Terry 2417 Como Avenue			BELL, B	RUCE F
St. Paul, MN 55108	-		ART UNIT	PAPER NUMBER
	•		1746	
			DATE MAILED: 09/30/200	3

### Notice of Fee Increase on October 1, 2003

If a reply to a "Notice of Allowance and Fee(s) Due" is filed in the Office on or after October 1, 2003, then the amount due will be higher than that set forth in the "Notice of Allowance and Fee(s) Due" since there will be an increase in fees effective on October 1, 2003. See Revision of Patent Fees for Fiscal Year 2004; Final Rule, 68 Fed. Reg. 41532, 41533, 41534 (July 14, 2003).

The current fee schedule is accessible from (http://www.uspto.gov/main/howtofees.htm).

If the fee paid is the amount shown on the "Notice of Allowance and Fee(s) Due" but not the correct amount in view of the fee increase, a "Notice of Pay Balance of Issue Fee" will be mailed to applicant. In order to avoid processing delays associated with mailing of a "Notice of Pay Balance of Issue Fee," if the response to the Notice of Allowance is to be filed on or after October 1, 2003 (or mailed with a certificate of mailing on or after October 1, 2003), the issue fee paid should be the fee that is required at the time the fee is paid. If the issue fee was previously paid, and the response to the "Notice of Allowance and Fee(s) Due" includes a request to apply a previously-paid issue fee to the issue fee now due, then the difference between the issue fee amount at the time the response is filed and the previously-paid issue fee should be paid. See Manual of Patent Examining Procedure, Section 1308.01 (Eighth Edition, August 2001).

Effective October 1, 2003, 37 CFR 1.18 is amended by revising paragraphs (a) through (c) to read as set forth below.

Section 1.18 Patent post allowance (including issue) fees.

(a) Issue fee for issuing each original or reissue patent,

except a design or plant patent:	
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By a small entity (Sec. 1.27(a))	\$320.00
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Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at (703) 305-8283.

Page 4 of 4

PTOL-85 (Rev. 08/03) Approved for use through 04/30/2004.

JA186

	Application No.	Applicant(s)
	10/272 017	
Notice of Allowability	10/372,017 Examiner	SENKIW, JAMES ANDRÉW
	Bruce F. Bell	1746
The MAILING DATE of this communication I claims being allowable, PROSECUTION ON THE MERI erewith (or previously mailed), a Notice of Allowance (PTC OTICE OF ALLOWABILITY IS NOT A GRANT OF PATE the Office or upon petition by the applicant. See 37 CFR	TS IS (OR REMAINS) CLOSED in DL-85) or other appropriate commi INT RIGHTS. This application is s	n this application. If not included unication will be mailed in due course. THI
This communication is responsive to the application		
The allowed claim(s) is/are <u>1-14</u> .		
The drawings filed on <u>21 February 2003</u> are accepted	d by the Examiner.	
Acknowledgment is made of a claim for foreign prior	ity under 35 U.S.C. § 119(a)-(d) o	r (f).
a) 🔲 All b) 🛄 Some* c) 🛄 None of the:	•	
1. 🔲 Certified copies of the priority documents	s have been received.	
2.  Certified copies of the prionty documents	s have been received in Application	on No
3.  Copies of the certified copies of the prior	ity documents have been receive	d in this national stage application from the
International Bureau (PCT Rule 17.2)	a)).	. *
* Certified copies not received:		
Acknowledgment is made of a claim for domestic price	prity under 35 U.S.C. § 119(e) (to	a provisional application).
(a) 🗌 The translation of the foreign language provisi	onal application has been receive	d.
Acknowledgment is made of a claim for domestic price	ority under 35 U.S.C. §§ 120 and/o	or 121.
oplicant has THREE MONTHS FROM THE "MAILING DA' elow. Failure to timely comply will result in ABANDONMEI A SUBSTITUTE OATH OR DECLARATION must be FORMAL PATENT APPLICATION (PTO-152) which give	NT of this application. THIS THR submitted. Note the attached EX.	EE-MONTH PERIOD IS NOT EXTENDAE
CORRECTED DRAWINGS must be submitted.		
(a) including changes required by the Notice of Dra	ftsperson's Patent Drawing Revie	w (PTO-948) attached
1) 🔲 hereto or 2) 🔲 to Paper No		
(b) including changes required by the proposed dra	wing correction filed, whic	ch has been approved by the Examiner.
(c) including changes required by the attached Exa	-	
Identifying indicia such as the application number (see 37 each sheet.	CFR 1.84(c)) should be written on th	he drawings in the front (not the back) of
DEPOSIT OF and/or INFORMATION about the tached Examiner's comment regarding REQUIREMENT F		
ttachment(s)		
X Notice of References Cited (PTO-892) ☐ Notice of Draftperson's Patent Drawing Review (PTO-9	48) 4 Interview	f Informal Patent Application (PTO-152) v Summary (PTO-413), Paper No
Information Disclosure Statements (PTO-1449), Paper		er's Amendment/Comment
Examiner's Comment Regarding Requirement for Depo of Rielagical Material		er's Statement of Reasons for Allowance
of Biological Material	9 Other	•

Application/Control Number: 10/372,017 Art Unit: 1746 Page 2

## **REASONS FOR ALLOWANCE**

1. The following is an examiner's statement of reasons for allowance:

The prior art of record fails to teach and/or suggest the key to applicants instant invention which is the critical distance between the anode and cathode in the generation of microbubbles and nanobubbles. The closest prior art of Zappi et al (6,315,886) shows an interelectrode gap of less than 0.00435 inches, whereas the applicants' instant critical distance is set to be between 0.005 to 0.140 inches. The prior art of Zappi et al discloses that their electrolyte is fed to the electrode stack under pressure where as the instant invention is not. Further, the Zappi et al patent talks about an insulation layer of bubbles, but never states whether they are nanobubbles or microbubbles or a combination thereof, or even if they will form at all. Applicants' have defined there critical distance to be the formation of nanobubbles and microbubbles in the electrode stack and since the prior art of Zappi et al does not disclose whether their invention yields both or not and because the interelectrode gap is smaller than applicants' interelectrode gap, it appears that the Zappi et al patent does not teach and/or suggest the emitter as set forth by the applicant's in their instant invention as now claimed. Therefore, the instant invention appears to be novel.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA188** 

Application/Control Number: 10/372,017 Art Unit: 1746

Page 3

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Bruce F. Bell whose telephone number is 703-308-2527. The examiner can normally be reached on Monday-Friday 6:30 AM - 3:00 PM.

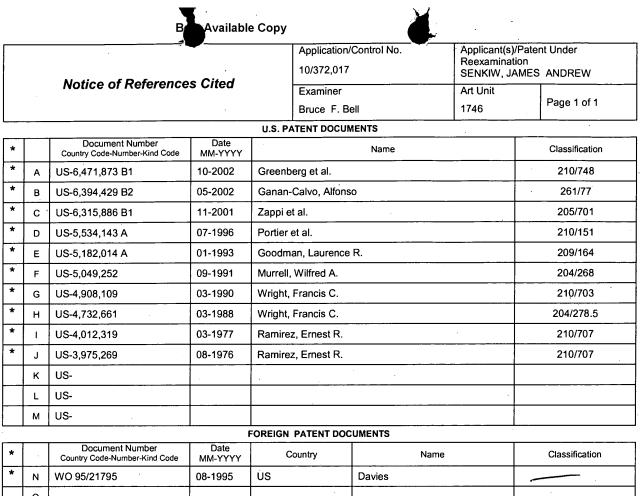
If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Randy Gulakowski can be reached on 703-308-4333. The fax phone number for the organization where this application or proceeding is assigned is (703) 872-9306.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-0661.

BFB September 29, 2003

Bruce F. Bell

Bruce F. Bell Primary Examiner Art Unit 1746



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NON-PATENT DOCUMENTS									
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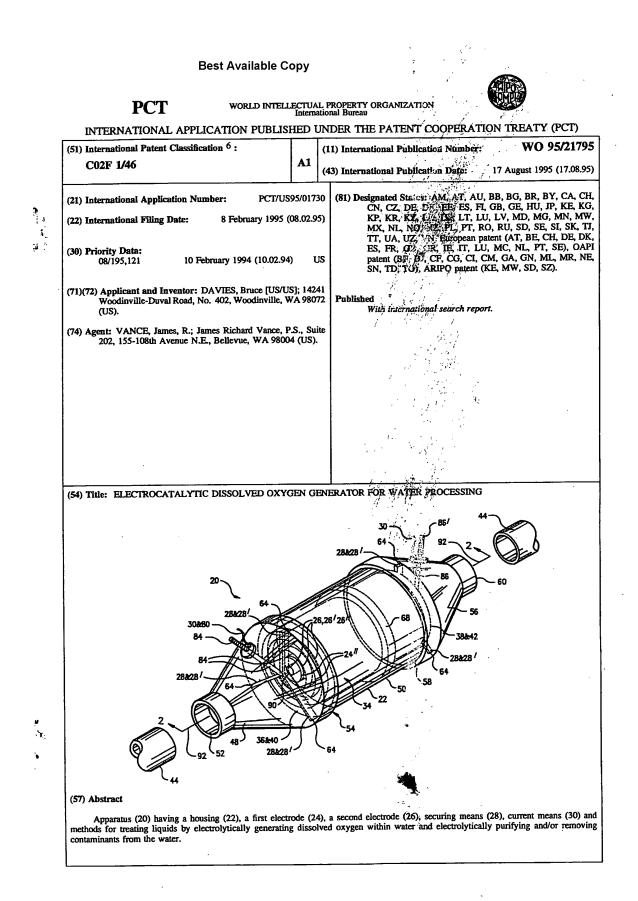
U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

Notice of References Cited

Part of Paper No. 4

JA190

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 193 of 1320



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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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-1-

WO 95/21795

PCT/US95/01730

5 Title: ELECTROCATALYTIC DISSOLVED OXYGEN GENERATOR FOR WATER PROCESSING

### COPYRIGHT NOTICE

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#### TECHNICAL FIELD

This invention relates to apparatus and methods for electrolytically treating liquids. More particularly, this 20 invention relates to apparatus and methods for generating dissolved oxygen within water and for electrolytically purifying and/or removing contaminants from the water.

#### BACKGROUND ART

A widely recognized problem within open and closed 25 hydraulic systems is the depletion or degradation of dissolved oxygen within water. This problem is particularly significant within environments that are highly dependant upon dissolved oxygen. For example, large and small bodies

JA193

PCT/US95/01730

-2-

of water that support aquatic and marine life require the generation and maintenance of large amounts of dissolved oxygen. Dissolved oxygen is a necessary requirement for the survival of aquatic organisms living in lakes, rivers, and

- 5 other bodies of water. Fish will die if the dissolved oxygen level drops below a given point. Persons familiar with aquatic and marine life often measure the quality and health of a body of water by the amount of dissolved oxygen present.
- Similarly, certain processes such as those used within 10 paper processing plants, water purification plants, and sewage treatment plants require the generation of large amounts of dissolved oxygen.

Various attempts have been made to aerate such bodies of water and, thereby, increase the amount of dissolved oxygen

15 contained therein. For example, air or gaseous oxygen have been forced under water and allowed to escape and bubble to the surface in an attempt to increase the level of dissolved oxygen within water. The main problem with gaseous oxygen, however, is that it is difficult to dissolve into water above

20 a given saturation point.

Other attempts to encourage oxygen entry into water include spraying the water into the air, mechanically splashing the surface of the water, and subjecting the water to intense elevated pressures within a pressurized container.

25 Mechanical methods of spraying the water into the air or mechanically splashing the water are very inefficient methods to produce dissolved oxygen. Furthermore, such mechanical methods are unable to reach higher levels of dissolved oxygen

JA194

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 197 of 1320

WO 95/21795

PCT/US95/01730

-3-

with any degree of stability.

In an unrelated area of technology, electrolysis has been used to break apart the various molecules of H<sub>2</sub>O or water to produce gaseous hydrogen and gaseous oxygen. It is 5 important to note, however, the significant difference between gaseous oxygen and dissolved oxygen.

The inventor has dedicated much of his life to the study of open and closed hydraulic systems. On April 17, 1990, U.S. Letters Patent No. 4,917,782, issued in the name of the 10 inventor for an electrolytic liquid purification process and

- apparatus. The apparatus and processes disclosed within U.S. Letters Patent No. 4,917,782 are significantly different from those of the present invention. Other disclosures that were considered in the prosecution of that patent include: Hughes,
- 15 Jr. et al. (U.S. Letters Patent No. 2,864,750; issued December 16, 1958); Mehl (U.S. Letters Patent No. 3,523,891; issued August 11, 1970); Doevenspeck (U.S. Letters Patent No. 3,679,556; issued July 25, 1972); Preis et al. (U.S. Letters Patent No. 3,728,245; issued April 17, 1973); Cassanovas et
- al. (U.S. Letters Patent No. 3,835,018; issued September 10, 1974); Phipps (U.S. Letters Patent No. 3,865,710; issued February 11, 1975); Okert (U.S. Letters Patent No. 3,925,176; issued December 9, 1975); Frame (U.S. Letters Patent No. 4,419,206; issued December 6, 1983); Neymeyer (U.S. Letters
  Patent No. 4,425,216; issued January 10, 1984); Branchick et al. (U.S. Letters Patent No. 4,436,601; issued March 13, 1984); Paniagua (U.S. Letters Patent No. 4,572,775; issued February 25, 1986); and Umehara (U.S. Letters Patent No.

JA195

PCT/US95/01730

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-4-

4,623,436; issued November 18, 1986).

The inventor believes that the listed disclosures taken alone or in combination neither anticipate nor render obvious the present invention. The cited disclosures do not 5 constitute an admission that such are relevant or material to the present claim. Bother the second

the present claims. Rather, the aforementioned disclosures relate only to the general field of the invention and are cited as constituting the closest art of which the inventor is aware.

10 DISCLOSURE OF INVENTION

The present invention comprises simple, easily used, inexpensive apparatus and methods for generating high levels of dissolved oxygen within water and for electrolytically purifying and/or removing contaminants from the water in an

- 15 environmentally safe fashion. The invention is compact, unobtrusive, functional, efficient, reliable, reusable, durable, rugged, easily constructed, inexpensive and economical to manufacture, and is easily installed and removed if needed. Minimal installation and access room is
- 20 needed. A minimum amount of manipulation is required for installation. Once installed, the invention is extremely simple to use and maintain. The apparatus of the invention does not require very much space. Consequently, replacement parts also require a minimal amount of storage space. The
- 25 present invention significantly increases the speed, simplifies the procedure, and dramatically enhances the efficiency of elevating the oxygenation level within a body of water.

JA196

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 199 of 1320

WO 95/21795

PCT/US95/01730

-5-

In addition to the foregoing benefits and other advantages that will be described further below, the present invention also overcomes all of the previously mentioned disadvantages of apparatus and processes heretofore used for

- 5 the stated purpose. For example, a significant benefit of the present invention is that it can be used within a closed, hydraulic conduit or pipeline. The present invention eliminates the need for bulky, expensive, complex, and maintenance intensive surface spray and mechanical aerators.
- 10 No longer must a business enterprise or municipality purchase large tracks of land for aeration, settlement, and treatment ponds. The aeration or dissolved oxygen generation process no longer must be exposed to surface air which creates aromatic, zoning, and ecology related problems.
- Installation of the invention does not mandate an 15 extensive capital investment that would otherwise be required. The cost to maintain the invention is insignificant in comparison to the expense of building and maintaining the type of commercial aeration projects now in
- 20 use. The savings to commercial enterprises by using this invention also includes not having to hire, pay, and contribute fringe benefits to numerous support personnel. The dangers of falling into open ponds and catching a finger, hand, limb, or clothing in operating machinery are also 25 eliminated.

To accomplish the aforementioned objectives, the present invention uses electrolysis processes to break apart water molecules and cause oxygen, created thereby, to go directly

> **OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

**JA197** 

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 200 of 1320

WO 95/21795

PCT/US95/01730

-6-

into a dissolved state. Due to the catalytic properties and action of the invention, the oxygen molecules do not pass through a gaseous state but rather are forced to assume an immediate dissolved state throughout the conversion process.

5 The oxygen, in effect, bypasses the gaseous stage that is otherwise required by other processes.

The resulting dissolved oxygen levels are relatively stable due to the cell design and do not require pressurization during the process. In addition, the 10 dissolved oxygen generating abilities of the invention are relatively unaffected by the presence of minerals, chemicals or organic materials that are present within the water. In fact, the invention scrubs or removes such contaminants from the water. The cell is noncontaminating and does not 15 introduce any additional metal, mineral, or chemical

components into the water.

The invention represents a dramatic and significant improvement over the electrolysis-type processes disclosed in the inventor's earlier granted patent (U.S. Letters Patent 20 No. 4,917,782). There is no need to precondition the water, as is usually required in other electrolysis-type processes. There is no need that particular minerals be present in the water for operation of the processes. In addition, water passing through the apparatus is not further contaminated by 25 the process, as commonly occurs during other electrolysis-

type processes where untreated metal electrodes dissolve.

The inventor knows of no other apparatus, wherein apparatus using an electrolysis process has been solely

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA198** 

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 201 of 1320

WO 95/21795

PCT/US95/01730

-7-

designed for the production of dissolved oxygen within water. Some electrolysis processes do produce minimal amounts of dissolved oxygen as a byproduct. However, such processes are unable to produce the higher levels of dissolved oxygen that 5 can be achieved by using the present invention.

Heretofore, using unprocessed water at 30 degrees centigrade such mechanical devices and/or processes could only obtain a maximum level of dissolved oxygen concentration of 7.6 parts per million (ppm). Processed water at 30 10 degrees centigrade could contain a maximum level of dissolved oxygen concentration of 10 parts per million (ppm) before reaching an insurmountable saturation point.

In comparison, unprocessed water at 30 degrees centigrade that is passed through the present invention can 15 readily obtain a dissolved oxygen concentration level of 20 parts per million.

Another method that has been used to generate elevated concentrations of dissolved gases is exposing the water or fluid to excessive gas pressurization. For example,

20 carbonated water or soda generally uses pressurization to obtain higher levels of gas saturation.

The elevated dissolved oxygen levels obtained through the present invention, however, are not obtained through the use of carbonation or elevated pressures. The water is not 25 carbonated. The aforementioned results are accomplished with no significant additional pressurization. The lack of pressurization is important to note. The processes used within the present invention in effect change the gas

JA199

PCT/US95/01730

-8-

balances of the water. In other words, the present invention rearranges the various gas percentages of oxygen, nitrogen, and contaminants within the water.

Henry's law generally states: The concentration of a <sup>5</sup> gaseous solute in a solution,  $C_g$ , is directly proportional to the partial pressure,  $p_g$ , of the gas above the solution. The resulting equation is  $C_g = k_g p_g$ , wherein  $k_g$  represents Henry's law proportionality constant. For example, at 25 degrees Celsius, oxygen gas collected over water at a total pressure 10 of 1.00 atmosphere (atm) is soluble to the extent of 0.0393

grams per liter.

Accordingly, if the altitude and temperature of an open body of water are known, one can determine the level of dissolved oxygen within the water by using Henry's law.

- 15 Henry's law, however, assumes that the percentage of gas contained within the water will be the same as that of the surrounding atmospheric air, or in other words seventy-eight percent (78%) nitrogen and twenty-one and nine-tenths percent (21.9%) oxygen.
- 20 The inventor has discovered that by electrocatalytically forcing oxygen within the water into a dissolved state, the apparatus of the present invention can create an unstable or quasi-stable condition. In effect, the dissolved gases contained within the water can exceed the heretofore believed 25 absolute saturation limit of one hundred percent (100%).

During the electrocatalytic process, some of the dissolved oxygen within the water evaporates, or bleeds or boils off, along with a corresponding amount of nitrogen. As

JA200

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 203 of 1320

WO 95/21795

PCT/US95/01730

-9-

a result, an almost stable condition is obtained. At this point the one hundred percent (100%) dissolved gas limit is not yet exceeded. However, since nitrogen has been purged or expelled from the system, the water can directly and

- 5 immediately absorb a comparable amount of dissolved oxygen without the oxygen having to enter a gaseous state. Thus, the concentration of dissolved oxygen can be increased without a similar increase in dissolved nitrogen. The oxygen level increases, and the nitrogen level decreases.
- 10 Consequently, the present invention allows one to reach triple saturation very easily and not violate Henry's law. The amount of gas in the solution remains at 100% to 101%, but processed repetitively, most if not all of such gas will be oxygen.
- 15 The apparatus is preferably placed within systems that recirculate water. Every time the water passes through the apparatus, more nitrogen is displaced and the level of dissolved oxygen increases. This process can continue until approximately twenty-two (22) parts per million of dissolved 20 oxygen are achieved and maintained, regardless of the
- temperature of the water, as long as the water temperature is below fifty (50) degrees Celsius. For example, the present invention can achieve and maintain a concentration of twentytwo (22) parts per million of dissolved oxygen all day long 25 when the water was at thirty (30) degrees Celsius.

A conventional use of Henry's law, however, would generally limit the concentration amount dissolved oxygen to around six (6) or (7) parts per million. In other words,

JA201

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 204 of 1320

WO 95/21795

PCT/US95/01730

-10-

according to Henry's law, the given body of water should have a dissolved oxygen concentration level of around 6 parts per million, but by using this invention the concentration level may be maintained at about 22 parts per million.

- 5 When trying to aerate water by pumping or blowing air underwater and then allowing the gas to bubble to the surface, the injected air comprises approximately seventyeight percent (78%) of nitrogen and twenty-two percent (22%) of oxygen. The relative percentage of absorption of such
- 10 gases is comparable to the respective amounts of gas injected below the water. Consequently, with the increase in dissolved oxygen, there is a significant (in excess of threefold) increase in the amount of dissolved nitrogen. Many fish and other aquatic life are sensitive to high 15 nitrogen levels and can become embolized if the nitrogen

levels are excessive.

In comparison, the level of dissolved oxygen can be effectively doubled, tripled, and even quadrupled in a single pass of water through the system without any absorption of

20 dissolved nitrogen. The results of this process far exceed that what can be achieved by using air blowing or other older techniques.

A different method to obtain dissolved oxygen is to first liquefy air to separate off the oxygen. The oxygen is 25 then stored in pressurized tanks for later injection back into the water reservoir. After being injected back into the water reservoir, the oxygen is allowed to again bubble back to the surface. Several problems with this method include

JA202

PCT/US95/01730

-11-

the added expense and difficulty to separate off the oxygen, and the cost and trouble to handle, store, and transport flammable pressurized oxygen tanks.

- Besides the significant reduction in cost, required equipment, and manual labor as compared to the previously mentioned pressurized method, the present invention generates dissolved oxygen. Unlike gaseous oxygen, dissolved oxygen is not explosive. Consequently, the present invention is much safer to use and operate. In addition, once the apparatus is
- 10 installed, the only needed component to operate the apparatus is a flow of water and a direct electrical current. It is usually much more convenient to obtain and maintain a supply of electricity than to keep replenishing a supply of pressurized oxygen tanks.
- 15 In effect, the inventor has discovered that by increasing the amount of dissolved oxygen in water, the oxygen molecules can actually displace and thereby remove or reduce the amount of dissolved minerals, oils, and organic matter contained in the water. Not only is healthier water
- 20 created, but the cleaning process does not use additional chemical additives to do so.

The inventor has actually created an electronically controlled scrubbing device that scrubs contaminated water at a molecular level.

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More particularly, the apparatus of the present invention can be used to treat water having physical, chemical, and/or biological contaminants. Although not a cure-all for every water problem, dissolved oxygen generation

JA203

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 206 of 1320

WO 95/21795

PCT/US95/01730

-12-

with electronic purification will help or cure contamination problems in most cases. The following explanation describes how the invention affects each type of contamination.

The electrical flow and field generated by the invention 5 causes a coagulation or lumping together of solids, colloids, and thin oils that comprise physical contaminants within water.

In addition, the electrolysis within the electrolytic or electrocatalytic cell of the invention adds large amounts of 10 dissolved oxygen to the water that in turn cause the oxidation and destruction of many other contaminants. As a result of the coagulation and oxidation just mentioned, once the water is allowed to settle, the contaminants easily fall or settle out of the solution.

Such coagulation, oxidation, and removal of settled contaminants eliminate algae, hydrogen sulfide, and other elements that create most obnoxious odors. For example, the presence of hydrogen sulfide generally creates a smell of raw sewage.

20 It is important to note that by using the electrocatalytic purification apparatus and processes described herein, such purification can be accomplished without using or adding other chemicals or agents to treat the water.

25 Chemical contaminants can also be reduced by subjecting the water to the high levels of dissolved oxygen. The dissolved oxygen oxidizes and breaks down many chemicals and hydrocarbons in low concentrations. Minerals and dissolved

JA204

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 207 of 1320

WO 95/21795

PCT/US95/01730

-13-

metals in the water also coagulate into filterable solids as do many soaps and phosphates.

Biological contamination can be safely and effectively treated using the present invention. It is important to note 5 that dissolved oxygen is an effective, natural bactericide that is not toxic to animals, fish, or plants.

Anaerobic bacteria live without oxygen. An example of a virulent type of anaerobic bacteria is that commonly found in stagnant water. The simple introduction of oxygen into 10 the water generally kills anaerobic bacteria.

Aerobic bacteria, which live with oxygen, can also be killed by introducing additional dissolved oxygen into the water. This is accomplished by raising the level of dissolved oxygen until the aerobic bacteria die. What

15 happens is that excess oxygen breaks down the outer wall of the bacteria cell causing the death of the organism. Consequently, to the inventor's knowledge no bacteria, not even the microscopic organisms, are immune to high levels of dissolved oxygen.

20 Because dissolved oxygen remains in the water, long pipe lengths and storage tanks can be cleansed and purified of contaminants by what is referred to as a residual kill attributed to dissolved oxygen.

Dissolved oxygen can also be used very effectively to 25 kill fecal coliform bacteria this is found in waters having sewage contamination. For example, many cities use oxygen to purify the water output being expelled from sewage plants. This is done because the introduction of oxygen is an

JA205

PCT/US95/01730

-14--

effective way to treat water without incurring harmful environmental side effects.

The noncontaminating nature and qualities of this invention make the apparatus particularly valuable for use 5 with fish farms, lakes, drinking water, and other environmentally sensitive water reserves. The invention can similarly be used within aquatic, waste water treatment, sewage treatment, water purification, paper processing, and many other industries that require high levels of dissolved 10 oxygen within the water. Large open bodies of water can now be economically maintained having a stable, balanced,

supersaturated state of dissolved oxygen. The primary purpose of the invention, is not necessarily

the coagulation of contaminants, although this is an 15 extremely beneficial by-process. Instead, the primary purpose of the invention is to increase the level of dissolved oxygen within the water.

The present invention is a flow-through system. In other words, the invention allows the water to pass right

- 20 through the apparatus without having to force the water in or out of the apparatus. The system is preferably not serpentine in shape or operation. If the system was serpentine within the cell itself, turbulence would be introduced and the system would not work as well.
- 25 Consequently, the invention permits high water flow rates with only a minimum amount of drag or flow resistance. The rate of flow can and should be substantially constant.

JA206

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 209 of 1320

WO 95/21795

PCT/US95/01730

-15-

The invention can be installed into and will not interfere with most water pumping and storage systems.

The construction and materials used within the invention make the apparatus and processes used nearly, if not 5 completely, immune to normal water pressure and temperature changes.

To achieve the aforementioned general and specific objectives, the present invention generally comprises a housing, a first electrode or electrical conductor, a second 10 electrode or electrical conductor, securing means, and current means. Each of these elements and additional elements will be discussed in substantial detail below within the Best Mode For Carrying Out The Invention portion of the patent and are included herein by reference.

15 These and other objectives and advantages of the present invention will become more readily apparent upon reading the following disclosure and referring to the attached drawings.

## BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 is an isometric view of a preferred embodiment of 20 the invention illustrating a specially designed electrocatalytic cell, depicted in phantom lines, within an exterior housing.

FIG. 2 is an enlarged, cross-sectional, side-elevational view of the invention shown in Figure 1, further illustrating 25 placement and interconnection of the various components of the apparatus.

FIG. 3 is a partially exploded, isometric view of the invention illustrating the internal components of the

JA207

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 210 of 1320

WO 95/21795

PCT/US95/01730

-16-

electrocatalytic cell.

FIG. 4 is a transverse, cross-sectional view of the invention as seen within a plane defined by line 4-4 in Figure 2.

5 FIG. 5 is a schematic diagram showing various additional components that can be used with the apparatus to aerate, oxygenate, and treat an open aquatic or marine pond, such as a fish pond.

FIG. 6 is a schematic diagram of an alternative form of 10 putting the invention into practice to aerate, oxygenate, and treat waste water from a paper mill.

One should understand that the drawings are not necessarily to scale and the elements are sometimes illustrated by graphic symbols, phantom lines, diagrammatic

15 representations, and fragmentary views. In certain instances, the inventor may have omitted details which are not necessary for an understanding of the present invention or which render other details difficult to perceive.

## BEST MODE FOR CARRYING OUT THE INVENTION

- 20 Referring to the drawings, wherein like numerals indicate like parts, the present invention generally comprises an apparatus 20 having a housing 22, a first electrode 24, a second electrode 26, securing means 28, and current means 30.
- 25 The housing 22 defines an enclosure 32 within which an electrolytic or electrocatalytic cell 34 is contained. The housing 22 can have any desired cross-sectional configuration and be of any desired length. In the preferred embodiment of

JA208

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 211 of 1320

WO 95/21795

PCT/US95/01730

-17-

the invention, the housing 22 has an elongated, tubular, generally cylindrical shape. Rectangular, triangular, square, or other cross-sectional shapes may alternatively be used.

- 5 The inventor believes that the minimum exterior size of the housing 22 can be as small as a one (1) inch outer diameter. The inventor believes that the maximum exterior size of the housing 22 can be as large as a six (6) feet outer diameter.
- 10 The housing 22 has a first end 36 and an opposed second end 38. An inlet opening 40 or port is located at the first end 36. An outlet opening 42 or port is located at the opposed second end 38.

The housing 22 can be directly connected or secured to 15 a conduit or pipe 44 in any manner that permits the generally collinear passage of water 46 into the inlet opening 40, through the enclosure 32, and out of the housing 22 through the outlet opening 42.

The inventor prefers that the pipe 44 have a diameter of 20 about two (2) to three (3) inches. Of course, other sizes of pipe 44 can alternatively be used.

The housing 22 may be provided with an inlet coupling 48 that is connected between the pipe 44 and a main portion 50 of the housing 22 that encloses the cell 34. The inlet 25 coupling 48 is designed to provided a gradually increasing taper from an inlet end 52 to an opposed outlet end 54. The tapered design is intended to provide for a smooth flow of the water 46 through the apparatus 20. The inlet end 52 has

JA209

PCT/US95/01730

a narrower cross-sectional area or circumference as compared to the opposed outlet end 54. Similarly, the outlet end 54 has a larger cross-sectional area or circumference as compared to the opposed inlet end 52. The narrower inlet end 5 52 is sized and dimensioned to be secured to an opening

-18-

within the pipe 44.

The housing 22 could also be provided with an outlet coupling 56. The outlet coupling 56 is generally similar to the design of the inlet coupling 48. More particularly, the

- 10 outlet coupling 56 is connected between the pipe 44 and the main portion 50 of the housing 22 that encloses the cell 34. The outlet coupling 56 has an inlet end 58 and an opposed outlet end 60. The inlet end 58 of the outlet coupling 56 has a wider cross-sectional area or circumference as compared
- 15 to the opposed outlet end 60. Similarly, the outlet end 60 of the outlet coupling 56 has a narrower cross-sectional area or circumference as compared to the opposed inlet end 58. The narrower outlet end 60 is sized and dimensioned to be secured to an opening within the pipe 44.
- 20 The housing 22, inlet coupling 48, and outlet coupling 56 should be manufactured from a material that does not conduct electrical current. For example, it is preferred that the housing 22, inlet coupling 48, and outlet coupling 56 be manufactured from a PVC, Teflon, nylon, or any other nonconductive material. Of course, other materials could alternatively be used.

The joint or junctures between the inlet coupling 48, main portion 50 of the housing 22, and the outlet coupling 56

JA210

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 213 of 1320

WO 95/21795

PCT/US95/01730

-19-

may be sealed and secured by any appropriate means, such as by a threaded connection or use of an adhesive.

The cell 34 contains a plurality of bipolar, preferably tubular-shaped electrodes. The first electrode 24 and second electrode 26 are positioned or located within the enclosure defined by the main portion 50 of the housing 22 between the inlet opening 40 and outlet opening 42.

The first electrode 24 may comprise any desired shape or configuration that accomplishes the desired objectives. The 10 first electrode 24, however, should be capable of carrying an electrical charge and current.

The second electrode 26 may also comprise any desired shape or configuration that accomplishes the desired objectives. The second electrode 26 should also be capable 15 of carrying an electrical charge and current.

The first electrode 24 is juxtaposed near to the second electrode 26. However, there is a spaced relationship between the first electrode 24 and the second electrode 26. It is important to remember that the water 46 must be capable

- 20 of passing between the first electrode 24 and the second electrode 26 in a generally unimpeded manner, and an electrical charge and current must be capable of being passed through the water 46 from the first electrode 24 to the second electrode 26.
- 25 To achieve a fixed or adjustable spaced relationship between the first electrode 24 and the second electrode 26, securing means 28 that accomplish this task are provided. Such securing means 28 secure the first electrode 24 and the

JA211

PCT/US95/01730

-20-

second electrode 26 within the main portion 50 of the housing 22 in such a manner that the spaced relationship between the first electrode 24 and second electrode 26 can be easily maintained. The securing means 28 may take any desired form 5 or design that accomplishes the required task. One particular form for the securing means 28 will be discussed in more detail further below.

Although other configurations can be used with this invention, the interior of the cell 34 preferably consists of 10 two (2) or more coated tubular shaped catalytic electrodes 24 and 26 or curved plates. For example, in the preferred embodiment of the invention, the first electrode 24 comprises a plurality of elongated, coaxially-positioned, tubular plates 24'. For increase efficiency, the plates 24' of the first electrode 24 should have a generally collinear orientation with respect to passage of the water 46 through the enclosure 32.

In a similar manner, the second electrode 26 also comprises a plurality of elongated, coaxially-positioned, tubular plates 26'. The plates 26' of the second electrode 26 should also have a generally collinear orientation with respect to passage of the water 46 through the enclosure 32.

Since most hydraulic conduits or pipelines have a generally hollow, cylindrical shape, it is preferred that the 25 plates 24' and 26' of the first electrode 24 and second electrode 26 also comprise elongated, generally hollow, cylindrical shapes. When placed within the housing 22, the plates 24' and 26' should have successively larger diameters

JA212

PCT/US95/01730

-21-

so that they fit in a telescopic manner within one another.

Of course, other configurations of the plates 24' and 26' can alternatively be used, if desired. However, to minimize the amount of turbulence of the water 46 passing 5 through the enclosure 32, the inventor prefers to use plates 24' and 26' that have successively larger but similar crosssectional design as that of the pipe 44. The preferred configuration of the plates 24' and 26' is that of a hollow, cylindrical tube, pipe, or bent plate.

10 The plates 26' of the second electrode 26 are interposed between respective, proximate plates 24' of the first electrode 24. Although the present invention functions in a very different manner, the design is similar to that of a multi-plated capacitor using successively larger or smaller 15 spaced tubes placed within one another.

The spaced interposition of the plates 24' and 26' is important so that a closed electrical circuit can be created only when water 46 is passed between the plates 24' and 26'. Furthermore, with one exception that will be discussed 20 further below, it is desirable that the amount of surface area facing the respective interposed plates 24' and 26' be

maximized to better facilitate passage of current therefrom and affect a greater amount of water 46 passing therebetween.

Such interposition may be accomplished by using securing 25 means 28 that spaces the plates 26' of the second electrode 26 in an interposed relationship between the plates 24' of the first electrode 24. For example, the inventor prefers to use generally "Y"-shaped plate or electrode spacers, end

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA213

PCT/US95/01730

-22-

braces, or guides 28' to quickly and easily hold and space
the plates 24' and 26' relative to one another. The spacers
or guides 28' are adhered or otherwise secured to the plates
24' and 26'. A friction fit between the spacers or guides
5 28' and the plates 24' and 26' may be sufficient.
Alternatively, the spacers or guides 28' may be provided with
notches 62, channels, or indentations therein that are
designed to receive and retain the variously sized plates 24'
and/or 26'. The outermost edges 64 of the nonelectrically
10 conductive spacers or guides 28' may be braced or otherwise
secured to the interior sidewalls 66 of the housing 22.

The securing means 28 may further comprise means for preventing the passage of the water 46 through the enclosure 32 except between the first electrode 24 and the second 15 electrode 26. For example, a spacing or spacer ring 68 may be placed between the plates 24' and 26' and the interior sidewalls 66 of the main portion 50 of the housing 22.

To maximize the amount of retained dissolved oxygen within the water 46, the housing 22, the first electrode 24, 20 the second electrode 26, the securing means 28, and the current means 30 are all configured and dimensioned to reduce turbulence within the water 46 passing through the enclosure 32, thereby allowing the water 46 to maintain a maximum laminar flow.

25

To minimize turbulence, the inventor prefers to use a housing 22 that has the same general cross-sectional configuration as the pipe 44. In addition, after placement of the cell 34 within the housing 22, the remaining cross-

JA214

PCT/US95/01730

-23-

sectional area available for passage of the water 46 through the enclosure 32 should comprise approximately the same area, or generally comparable cross-sectional area, as that of the interior area of the pipe 44. As a result, there should be 5 no appreciable increase or decrease in the pressure within the water 46 as the water 46 passes through the enclosure 32.

The inventor desires to eliminate adverse venturi affects within the apparatus 20 and system. If a venturi affect occurs, there will be a lowering of water pressure. 10 If there is a pressure drop, gases will come out of the solution which is undesirable.

Consequently, the cross-sectional area of the apparatus 20 is enlarged, as compared to the input pipe 44. Such enlargement accounts for the added cross-sectional dimensions 15 of the internal components within the apparatus 20. The rate of flow of the water 46 through the system should not dramatically increase or decrease.

To further reduce turbulence within the enclosure 32, the inventor prefers to use plates 24' within the first 20 electrode 24 and plates 26' within the second electrode 26 that have a respective leading edge 24" and 26" that is tapered to a relatively sharp edge. Similarly, the trailing edge 24"' of the plate 24' should be tapered to a relatively sharp edge. The trailing edge 26"' of the plate 26' should 25 also be tapered to a relatively sharp edge. Tapering of the leading and trailing edges further reduce turbulence within the water 46.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 218 of 1320

WO 95/21795

PCT/US95/01730

-24-

The invention also contemplates the use of current means 30 for supplying a direct electrical current to the first electrode 24, through the water 46, and into the second electrode 26 to form a closed electrical circuit or loop. In 5 effect, the current means 30, the first electrode 24, the water 46, and the second electrode 26 define the electrolytic or electrocatalytic cell 34.

In the preferred embodiment of the invention, the current means 30 comprises an electrical power supply 80 or 10 source that is positioned externally from the housing 22 or cell 34. For example, a power supply 80 similar that described in U.S. Letters Patent No. 4,917,782 may be used with the present invention.

The first and second electrodes 24 and 26 are connected 15 to the power supply 80 and are thereby electrically active in the water 46 solution. In is important to note that each electrode 24 and 26 are set in electrically insulated material that insures proper spacing and minimum current leakage between the electrodes 24 and 26.

20 A very low voltage is used. For example, in a typical application the power supply 80 delivers about one (1) volt to sixty (60) volts of direct current to the plate 24' and 26' defining the cell 34. The actual amount of voltage will depend upon the conductivity, temperature, and elevation of 25 the water 46.

The cell 34 operates on about 0.5 amperes to 600 amperes depending upon the size and length of the first and second electrodes 24 and 26 (respectively comprising plates 24' and

JA216

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 219 of 1320

WO 95/21795

PCT/US95/01730

-25-

26'), and depending upon the characteristics of the water 46 such as its conductivity.

Electrical charges and currents within these ranges do not adversely effect fish.

5 In addition, the system uses an isolated direct current (DC) voltage system within the cell 34. Since the fish are not allowed to go through the cell 34, the fish are not adversely effected. The passage of the fish can be blocked by an input screen (not shown). Even if the input screen was 10 removed, the fish would not be able to pass through the filters and related equipment used with the cell 34. There is no voltage leakage outside of the cell 34 back to the pond at all. The transformer is isolated from the alternating current (AC) line and only director current (DC) is exposed 15 to the water 46. Consequently, the only electrical flow occurs within the cell 34 itself.

Normally, when water 46 is heated the amount of dissolved oxygen contained therein will necessarily decrease. Because of this problem, one would be motivated not to heat 20 the water 46. Since subjecting the water 46 to an electrical current will necessarily heat the water 46, commonly accepted

principles teach away from the present invention.

However, when water 46 is heated its conductivity actually increases. The increased conductivity of the water 25 46 counteracts any lowering of the total amount of dissolved gas contained within the water 46 due to the temperature Consequently, the processes of the present increase. invention can be effectuated without adverse impact from the

**JA217** 

PCT/US95/01730

-26-

water 46 becoming partially heated.

Since water is not as electrically conductive when cold, the present invention functions even better when the water is warmer or heated. At higher temperatures, the apparatus 20 5 works extremely well.

The first electrode 24 and the second electrode 26 are bipolar. Consequently, an electrical timer 82, similar to that described in U.S. Letters Patent No. 4,917,782, can be used to periodically reverse the polarity within the cell 34.

10 By reversing the polarity within the cell 34, debris adhering to the plates 24' and 26' can be urged to fall off.

The time periods for electrical current reversals vary depending upon the type and amount or level of water contamination and the debris buildup. The inventor prefers

15 a minimum reversal time period of about ten (10) minutes. A maximum reversal time period should be about eight (8) hours. Due to this electrically self-cleaning feature, no mechanical cleaning of the cell 34 is generally required.

The oxygen generation process described herein uses non-20 consumable catalytic type electrodes 24 and 26 and/or plates 24' and 26'. The spacing of the first and second electrodes 24 and 26 and shape of the cell 34 are designed to maximize or optimize dissolved oxygen production. The applied voltage and current levels are also designed to maximize production

25 of dissolved oxygen.

The spacing between respective plates 24' and 26' varies from about 0.1 inch to 1.0 inch depending on the type of water contamination that is being treated. The desired flow

JA218

PCT/US95/01730

-27-

rates, pipe size, and type of contamination being treated will primarily dictate the number of electrode plates 24' and 26' that must be used.

Power is supplied to the electrode plates 24' and 26' 5 positioned within the enclosure 32 by means of one or more cables, wires, and/or straps 84 and 86 that are welded or otherwise electrically connected or secured to plates 24' and 26', respectively. In the preferred embodiment, an electrically conductive strap 84 is provided for the set of 10 plates 24' that comprise first electrode 24. Similarly, an electrically conductive strap 86 is provided for the set of

plates 26' that comprise second electrode 26.

The strap 84 is connected in a parallel fashion to each plate 24' within the first set of electrodes 24. The strap 15 86 is connected in a parallel fashion to each plate 26' within the second set of electrodes 26.

The opposed terminal ends 84' and 86' of the straps 84 and 86 are past through the sidewall 66 of the housing 22, whereupon the terminal ends 84' and 86' are connected to the 20 external power supply 80 by any convenient manner. Thus configured, an electrical current can be past through the cell 34 once water 46 fills the gap between the respective plates 24' and 26'.

To reduce the amount of maintenance required by the 25 invention, the electrically conductive straps 84 and 86 are welded to each electrode plate 24' or 26' within that particular set of electrodes 24 or 26, respectively.

JA219

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 222 of 1320

WO 95/21795

#### PCT/US95/01730

-28-

If the straps 84 and 86 have any significant width thereto, a slot 88, channel, or notch may be cut into each electrode plate 24' or 26' to accommodate the placement of the straps 84 or 86 therein. As a result, the straps 84 and

5 86 may be mounted or placed edgewise within the flow path of the water 46 and thereby minimize its affect on turbulence within the water 46.

Within the preferred embodiment of the invention, a centermost portion of the cell 34 comprises a solid, thin, 10 cylindrical water block 90. Water block 90 may or may not be tapered to a point. The water block 90 is designed to further prevent water 46 from passing through the enclosure 32 without first passing between two oppositely charged electrode plates 24' and 26' and thereby being 15 electrocatalytically treated.

The reader is reminded of the above-mentioned spacing or spacer ring 68, that is placed between the plates 24' and/or 26' and the interior sidewalls 66 of the main portion 50 of the housing 22. That spacing or spacer ring 68 also prevents

20 the passage of the water 46 through the enclosure 32 except between the first set of plates 24' that define the first electrode 24 and the second set of plates 26' that define the second electrode 26. Thus positioned, water 46 cannot bypass the cell 34 unless a different bypass pipe (not shown) is 25 used.

The centermost cylindrical water block 90, the outermost spacing or spacer ring 68, the plate or electrode spacers or guides 28', the inlet coupling 48, the outlet coupling 56,

JA220

PCT/US95/01730

-29-

and the interior sidewalls 66 of the housing 22 are all preferably manufactured from an electrically nonconductive material such as, but not limited to, PVC, Teflon, or nylon.

- To facilitate proper welding of the electrical straps 84 5 and 86 to the appropriate sets of plates 24' and/or 26', both the straps 84 and 86 and both sets of plates 24' and 26' or electrodes 24 and 26 are preferably manufactured from titanium. Of course, other metals may also be used, but the inventor prefers to use titanium.
- In addition, either or both of the sets of plates 24' and/or 26', such as the first electrode 24 and/or the second electrode 26, are coated with iridium oxide, ruthenium oxide, rhodium oxide, palladium oxide, osmium oxide, platinum oxide, or any other coating material that enhances the capability of
- 15 the cell 34 to perform its function. The inventor, however, has found that the particularly selected metals and coatings used for the cell 34 as stated herein have unique catalytic properties.

To further enhance the efficiency of the cell 34, the 20 leading edge 24" of each plate 24' within the first electrode 24 is set forward of or set backward of each proximate leading edge 26" of each plate 26' within the second electrode 26. The aforementioned spacing along the longitudinal axis 92 of the cell 34, as determined by the 25 passage of water 46 through the enclosure 32, should be about one to one and one-half inches (1" to 1-1/2") between the leading edges 24" of the plates 24' of the first electrode 24 and the leading edges 26" of the plates 26' of the second

JA221

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 224 of 1320

WO 95/21795

PCT/US95/01730

-30-

electrode 26. In other words, the leading edges 24" of the plates 24' forming the first electrode 24 should not be aligned with each proximately positioned leading edge 26" of the plates 26' forming the second electrode 26. This

- 5 misalignment is intended to make sure that the plates 26' are not within the electrical flow that supplies power to plates 24'. Similarly, the plates 24' should not be within the electrical flow that supplies power to plates 26'.
- Furthermore, the leading edges 24" and 26" and the 10 trailing edges 24"' and 26"' of plates 24' and 26" should not be actively involved within the electrolysis process or excessive wear may occur.

The present invention may be practiced without the use of a mechanical filter. In effect the invention can be practiced without the use of consumable chemicals, filters, or other related equipment. No chemicals are used in this process other than for ph adjustment.

Alternatively, if desired, one or more filters 94 may be operatively connected to the housing 22 or be placed upstream 20 and/or downstream from the apparatus 20 within the pipe 44.

- For example, the filter 94 may remove contaminants from the water 46 before the water 46 passes through the enclosure 32. Figure 5 illustrates the filter 94 installed upstream of the apparatus 20. The filter 94 may take any desired form.
- 25 However, the filter 94 should not significantly retard the flow of water 46 through the apparatus 20.

The water 46 may be gravity fed through the pipe 44 and through the apparatus 20.

JA222

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 225 of 1320

WO 95/21795

PCT/US95/01730

-31-

To prevent excessive build-up within the holding tank 100, the inventor uses a flood sensor (not shown) that governs the input of waste water 46' through the input pipe 44.

5

Alternatively, one or more pumps 96 may be used to increase the pressure of the water 46 and thereby urge the water 46 to pass through the enclosure 32. The pump or pumps 96 can be operatively connected to the apparatus 20 or to the pipe 44, or actually be integrally formed within the housing 10 22 of the apparatus 20.

The invention is intended for use with water circulation apparatus to clean and purify the water 46 and remove the minerals therefrom. Prior to passing through the apparatus 20 and/or after having passed through the apparatus 20, the

15 water 46 may be deposited into a holding pond 98, holding tank 100, or the like. Figure 5 schematically illustrates the use of the holding pond 98, such as an open aquarium, fish pond, waste water treatment pond, aquarium, hot tub, or swimming pool.

20 Figure 6 schematically illustrates the use of the apparatus 20 with one or more enclosed holding tanks 100. In essence, the holding tank 100 is operatively connected to an input pipe 102. The holding tank 100 is capable of holding or detaining the water 46 after the water 46 has past through 25 the enclosure 32, thereby permitting contaminants within the water 46 to settle to a bottom 104 of the holding tank.

.To achieve this end, contaminated water 46 is initially pumped into the holding tank 100 through input pipe 102 where

> **OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

PCT/US95/01730

-32-

the water 46 is stored. Each holding tank 100 is provided with an inlet port 106 and an outlet port 108. The inlet port 106 is positioned near a lower portion 110 of the holding tank 100 to receive unprocessed water 46' 5 therethrough. The outlet port 108 is positioned near an upper portion 112 of the holding tank 100 to permit removal of processed water 46" therethrough.

Some of the contaminants may be mechanically removed from the unprocessed water 46' by means of one or more 10 filters 94 that are placed within the input pipe 102.

Once passed into the holding tank 100 through the inlet port 106, some of the contaminants may settle out of the unprocessed water 46' before the water 46 is past into the cell 34.

15 The unprocessed water 46' is drawn from near the bottom 104 or middle portion 114 of the holding tank 100 and is pumped into the cell 34 where the water 46 is electrocatalytically scrubbed. After exiting the cell 34, the processed water 46" is pumped back into the holding tank

20 100 whereupon the coagulated contaminants can settle to the bottom 104 of the holding tank 100.

Due to the high electrical flow between the plates 24' and 26', high dissolved oxygen levels ionize and coagulate the solids within the water 46 thereby causing such solids to

25 precipitate. The oxidized solid waste can then be removed from the bottom 104 of the holding tank 100. Hydrogensulfide, phenols, and trace oils can all be broken down by using this method. In particular, when BOD and COD have

JA224

PCT/US95/01730

-33-

their oxygen demand met and then exceeded by a large amount, the breakdown of the waste is very rapid.

In comparison, the generation of dissolved oxygen using other technologies is very slow, from hours into days and 5 weeks, due to an inability to effectively produce high dissolved oxygen levels.

With the present invention, however, the cell 34 is specially designed to maximize the production of dissolved oxygen in water 46 flowing through it. The shape of the 10 parts of the apparatus 20 are designed to reduce turbulence and to maintain a high level of dissolved oxygen within the water 46. Furthermore, the selection of voltage and current levels are also set to maximize dissolved oxygen generation.

With each successive pass through the cell 34, the water 15 46 is further purified. In addition, with each pass nitrogen gas is being expelled from the processed water 46" and is being immediately replaced with dissolved oxygen that is created during the electrocatalytic process.

The holding tank 100 preferably has a vent 116 located 20 near its uppermost portion 118. Gaseous nitrogen, oxygen and/or other undesired gases that are expelled from the cell 34 will float to the upper surface 120 of the holding tank 100, whereupon such gases can be vented, released, or expelled to another holding tank 101' for further processing 25 or be vented to the outside atmosphere. The venting of such gases eliminates the possibility that such gases will be reabsorbed back into the processed water 46".

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 228 of 1320

WO 95/21795

PCT/US95/01730

#### -34-

As a consequence of using the disclosed apparatus 20 and processes, processed, purified water 46" will naturally migrate to the upper portion 112 of the holding tank 100.

- In effect, a stratification of the water 46 occurs. As the 5 water 46 becomes more gasified or has less contaminants therein, such processed water 46" rises upward. Unprocessed water 46' containing contaminants that are clumped together fall to the bottom. Consequently, natural stratification occurs. Eventually the processed water 46" floats to the top 10 of the holding tank 100 and unprocessed or less processed
- water 46' falls to a lower level for reprocessing.

The apparatus 20, and particularly the holding tank 100, may also be provided with means for removing settled contaminants from the bottom 104 of the holding tank 100.

15 Any appropriate means to accomplish this task may be used.

The processed water 46" that remains near the upper surface 120 of the holding tank 100 can be drawn off by gravity or be pumped off into another holding tank 100', whereupon the process is again repeated.

- The present invention can be used without a holding tank 100 (as shown in Figure 5), with a single holding tank 100, or with a plurality of holding tanks 100, 100' and 100". At each successive level or passage to the next holding tank 100, 100' and 100", the processed water 46" will become 25 progressively purified of contaminants will be
- 25 progressively purified of contaminants, will have significant amounts of dissolved nitrogen displaced therefrom, and will eventually have extremely high volumes of dissolved oxygen contained therein.

JA226

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 229 of 1320

WO 95/21795

PCT/US95/01730

-35-

As seen in Figure 6, a cell 34 is not installed within the third or last illustrated holding tank 100". By the time the water 46 reaches the third tank 100", the levels of dissolved oxygen contained within the water 46 are very high.

5 Consequently, the advantageous effects imparted by high levels of dissolved oxygen will continue even after the production of dissolved oxygen ceases. The inventor refers to this as a residual kill process where contaminants are continuing to be purged from the water reservoir within 10 holding tank 100".

In comparison, if only ozone is introduced into the water 46, fifteen seconds down the pipe 44 the ozone has dissipated and is no longer effective. If the present invention is used, a residual kill continues for an extended 15 period of time because, according to Henry's law, the balance

of dissolved gases within the water 46 must be maintained.

Chlorine may be placed within the water 46 and residual kill will occur for a substantial period of time. However, chlorine stays in the solution.

- Dissolved oxygen functions in a similar manner as chlorine but dissolved oxygen is eventually released into the atmosphere and the water solution is allowed to equalize. Thus, the present invention is much more ecologically friendly to the environment than using chlorine.
- 25

20

The present invention contemplates not only the apparatus 20 disclosed and claimed herein, in all of its forms and alternative embodiments, but also contemplates the described methods and processes for increasing the amount of

#### PCT/US95/01730

-36-

dissolved oxygen within water 46 and breaking down the molecular structure of water borne organisms.

Basically the process passes low direct current (DC) voltage, using a catalytic-type cell 34, through water 46.

5 The catalytic action of the cell 34 forces most of the oxygen that is broken apart from water molecules to go directly into a dissolved oxygen state.

The dissolved oxygen levels that can be created by these processes can increase a dissolved oxygen saturation point 10 from around three parts per million (3.0 ppm) to about twenty parts per million (20.0 ppm), depending on the starting conditions of the water 46. Under some circumstances, certain dissolved minerals react with the oxygen slowing down the oxygen generation process.

15 Such processes or methods may include the following steps:

First, passing water 46 through a conduit or pipe 44 into a housing 22 that defines an enclosure 32.

Second, passing the water 46 between a first electrode 20 24 and a second electrode 26 contained within the enclosure 32. The first electrode 24 and second electrode 26 each are capable of carrying an electrical charge and current. The first electrode 24 is juxtaposed near the second electrode 26 in a spaced relationship to the second electrode 26.

25 Third, supplying a direct electrical charge and current to the first electrode 24 so that the direct electrical current passes through the first electrode 24, through the water 46, and into the second electrode 26 to form a closed

JA228

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 231 of 1320

WO 95/21795

PCT/US95/01730

-37-

electrical circuit or loop. The means for supplying the direct electrical current, the first electrode 24, the water 46, and the second electrode 26, in fact, define an electrolytic and/or electrocatalytic cell 34. The direct
5 electrical current should be sufficient, when passed through the water 46, to break apart water molecules. The direct electrical current should also be sufficient to break down the molecular structure of water borne organisms and/or chemicals by means of electrolysis and by oxidization created
10 from the breaking apart of the water molecules.

#### Example

A small pond having a temperature of 30 degrees Celsius and a medium fish loading would typically have a dissolved oxygen saturation point of 7.6 parts-per-million (ppm). If 15 the fish loading or containment is higher, the dissolved oxygen saturation point would be slightly less than 7.6 ppm. When using other technologies, such as with air blowers as described above, the dissolved oxygen saturation point of 7.6 ppm can be maintained. Under perfect conditions, using 20 such apparatus might obtain a maximum saturation point of around 9.0 ppm. A higher amount of dissolved oxygen would result in healthier fish with better food conversion ratios. Use of air blowers, however, requires a substantial

amount of electrical power. For example, a typical rate of 25 power usage would be approximately 5.0 kilowatts per hour.

Using the apparatus of the present invention, the very same pond can be easily maintained with a dissolved oxygen saturation point of 14.0 to 16.0 ppm. The power requirements

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 232 of 1320

#### WO 95/21795

#### PCT/US95/01730

-38-

to obtain such a dissolved oxygen saturation point would only be 0.9 kilowatts of electrical power per hour.

To accomplish this phenomenal improvement, the apparatus only needs to use eight (8) electrode plates and have a 5 diameter of three (3) inches. The power requirement would be twenty-four (24) volts of direct current at thirty-seven (37) amperes. The flow rate of the treated water would be seventy (70) gallons per minute. The entire treatment system would consist of only the power supply, the cell, a filter, and a 10 pump for water movement.

It should be noted that the inventor has experimented with apparatus 20 having different cross-sectional configurations and has discovered that the design illustrated in the accompanying drawings can generate approximately

15 eighty percent (80%) more dissolved oxygen per watt of power than a system that uses flat, planar plates, as opposed to the cylindrical plates 24" and 26" discussed above.

The means and construction disclosed herein are by way of example and comprise primarily the preferred forms of putting the invention into effect. Although the drawings depict preferred and alternative embodiments of the invention, other embodiments have been described within the preceding text. One skilled in the art will appreciate that the disclosed device may have a wide variety of shapes and configurations. Additionally, persons skilled in the art to which the invention pertains might consider the foregoing teachings in making various modifications, other embodiments, and alternative forms of the invention.

JA230

#### PCT/US95/01730

-39-

It is, therefore, to be understood that the invention is not limited to the particular embodiments or specific features shown herein. To the contrary, the inventor claims invention in all of its forms, the including all 5 alternatives, modifications, equivalents, and alternative

embodiments that fall within the legitimate and valid scope of the appended claims, appropriately interpreted under the Doctrine of Equivalents.

## INDUSTRIAL APPLICABILITY

- The present invention may be utilized wherever simple, 10 reliable, easily used apparatus and methods are needed to increase the level of dissolved oxygen within a body of water. For example various aquatic, waste water treatment, sewage treatment, water purification, paper processing, and
- 15 many other plants and industries require high levels of dissolved oxygen within the water. The apparatus of this invention is compact, functional, unobtrusive, efficient, reusable, durable, rugged, is easily constructed, and is inexpensive and economical to manufacture.
- 20 The present invention has a special benefit of allowing its use with a wide variety of differently sized water conduits or pipelines. The apparatus may be easily manufactured with the appropriate length, width, and/or diameter to fit the needs of a particularly required 25 application.

The present invention may be secured to the conduit or pipeline in areas of extremely limited access. This feature makes the apparatus particularly unobtrusive and useful in

**JA231** 

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 234 of 1320

WO 95/21795

#### PCT/US95/01730

#### -40-

areas where aesthetics are important, such as with residential fish tanks.

Preexisting conduits or pipelines may be utilized. Other than to install the apparatus, there is no need to 5 modify, alter, or deface the preexisting machinery. Alternatively, the invention may be incorporated into or formed integrally within new hydraulic systems.

PCT/US95/01730

-41-

#### <u>CLAIMS</u>

What is claimed is:

Claim 1. Apparatus for increasing amount of dissolved 5 oxygen within water passing through a conduit or pipe, said apparatus comprising:

(a) a housing defining an enclosure therein, said housing having a first end and an opposed second end, said housing having an inlet opening at said first end, said
10 housing having an outlet opening at said second end, said housing capable of being secured to the conduit or pipe in a manner permitting the collinear passage of the water into said inlet opening, through said enclosure, and out of said outlet opening;

15 (b) a first electrode capable of carrying an electrical current positioned within said enclosure;

- (c) a second electrode capable of carrying an electrical current positioned within said enclosure, said first electrode being juxtaposed near said second electrode in
- 20 a spaced relationship to said second electrode, the water capable of passing between said first electrode and said second electrode;
  - (d) means for securing said first electrode and said second electrode in spaced relationship one to another within said enclosure; and
  - (e) means for supplying a direct electrical current to said first electrode, through the water, and into said second electrode to form a closed electrical circuit or loop,

JA233

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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PCT/US95/01730

-42-

said current means, said first electrode, the water, and said second electrode defining an electrolytic or electrocatalytic cell.

5 Claim 2. The apparatus of Claim 1, wherein said first electrode comprises a plurality of elongated, coaxiallypositioned, tubular plates, said plates of said first electrode having a generally collinear orientation with respect to passage of the water through said enclosure.

Claim 3. The apparatus of Claim 2, wherein said second electrode comprises a plurality of elongated, coaxiallypositioned, tubular plates, said plates of said second electrode having a generally collinear orientation with 15 respect to passage of the water through said enclosure, said plates of said second electrode being interposed between respective plates of said first electrode.

Claim 4. The apparatus of Claim 3, wherein said plates 20 of said first electrode comprise hollow cylindrical plates.

Claim 5. The apparatus of Claim 4, wherein said plates of said second electrode comprise hollow cylindrical plates.

25 Claim 6. The apparatus of Claim 3, wherein said securing means further comprises means for spacing said plates of said second electrode in an interposed relationship between said plates of said first electrode.

JA234

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 237 of 1320

WO 95/21795

PCT/US95/01730

#### -43-

Claim 7. The apparatus of Claim 6, wherein said plates of said first electrode and said plates of said second electrode each have a leading edge and a trailing edge, each said leading edge being tapered to reduce turbulence within 5 the water.

Claim 8. The apparatus of Claim 7, wherein each said trailing edge is tapered to further reduce turbulence within the water.

10

Claim 9. The apparatus of Claim 7, wherein each said leading edge of said first electrode is not aligned with each proximate said leading edge of said second electrode, each said leading edge of said first electrode being set forward 15 of or set backward of each proximate said leading edge of said second electrode.

Claim 10. The apparatus of Claim 1, wherein said securing means prevents passage of the water through said 20 enclosure except between said first electrode and said second electrode.

Claim 11. The apparatus of Claim 1, wherein said housing further comprises an inlet coupling having a narrower 25 inlet end and a larger outlet end, said narrower inlet end of said inlet coupling capable of being secured to the conduit or pipe.

JA235

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 238 of 1320

WO 95/21795

PCT/US95/01730

Claim 12. The apparatus of Claim 1, wherein said housing further comprises an outlet coupling having a larger inlet end and a narrower outlet end, said narrower outlet end of said outlet coupling capable of being secured to the 5 conduit or pipe.

-44-

Claim 13. The apparatus of Claim 1, wherein said housing, said first electrode, said second electrode, said securing means, and said current means are dimensioned to 10 reduce turbulence within the water and provide a passage for the water through said enclosure that has a generally comparable cross-sectional area to that of the conduit or pipe.

15 Claim 14. The apparatus of Claim 1, further comprising at least one holding tank operatively connected to the conduit or pipe, said holding tank capable of holding or detaining the water after the water has passed through said enclosure to permit contaminants within the water to settle 20 to a bottom of said holding tank.

Claim 15. The apparatus of Claim 14, wherein said holding tank further comprises means for removing contaminants that settle to said bottom of said holding tank.

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Claim 16. The apparatus of Claim 14, wherein said holding tank further comprises a vent through which undesired gases held within said holding tank may be released or

JA236

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 239 of 1320

WO 95/21795

PCT/US95/01730

-45-

expelled.

Claim 17. The apparatus of Claim 16, wherein said holding tank is provided with an inlet port and an outlet port, said inlet port being positioned near a lower portion of said holding tank to receive unprocessed water therethrough, said outlet port being positioned near an upper portion of said holding tank to permit removal of processed water therethrough.

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Claim 18. The apparatus of Claim 1, further comprising at least one pump operatively connected to the conduit or pipe, said pump urging the water to pass through said enclosure.

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Claim 19. The apparatus of Claim 1, further comprising at least one filter operatively connected to said housing or to the conduit or pipe, said filter removing contaminants from the water before the water passes through said 20 enclosure.

Claim 20. The apparatus of Claim 1, wherein said first electrode is coated with iridium oxide, ruthenium oxide, rhodium oxide, palladium oxide, osmium oxide, or platinum 25 oxide.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 240 of 1320

WO 95/21795

PCT/US95/01730

-46-

Claim 21. The apparatus of Claim 20, wherein said second electrode is coated with iridium oxide, ruthenium oxide, rhodium oxide, palladium oxide, osmium oxide, or platinum oxide.

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Claim 22. The apparatus of Claim 1, wherein said first electrode is manufactured from titanium.

Claim 23. The apparatus of Claim 22, wherein said 10 second electrode is manufactured from titanium.

Claim 24. A method for increasing the amount of dissolved oxygen within water and breaking down the molecular structure of water borne organisms, said method comprising 15 the steps of:

- (a) passing the water through a conduit or pipe into a housing defining an enclosure;
- (b) passing the water between a first electrode and a second electrode contained within the enclosure, the first electrode and the second electrode each capable of
- carrying an electrical current, the first electrode being juxtaposed near the second electrode in a spaced relationship to the second electrode;
- (c) supplying a direct electrical current to the first
   electrode so that the direct electrical current passes through the first electrode, through the water, and into the second electrode to form a closed electrical circuit or loop, the current means, the first electrode, the

JA238

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PCT/US95/01730

-47-

water, and the second electrode defining an electrolytic or electrocatalytic cell, the direct electrical current being sufficient when passed through the water to break apart water molecules, the direct electrical current further being sufficient to break down chemicals by means of electrolysis and by oxidization created from the breaking apart of the water molecules.

Claim 25. A method for increasing the amount of 10 dissolved oxygen within water and breaking down the molecular structure of water borne organisms, said method comprising the steps of:

- (a) passing the water through a conduit or pipe into a housing defining an enclosure;
- 15 (b) passing the water between a first electrode and a second electrode contained within the enclosure, the first electrode and the second electrode each capable of carrying an electrical current, the first electrode being juxtaposed near the second electrode in a spaced 20 relationship to the second electrode;
  - (C) supplying a direct electrical current to the first electrode so that the direct electrical current passes through the first electrode, through the water, and into the second electrode to form a closed electrical circuit or loop, the current means, the first electrode, the water, and the second electrode defining an electrolytic or electrocatalytic cell, the direct electrical current being sufficient when passed through the water to break

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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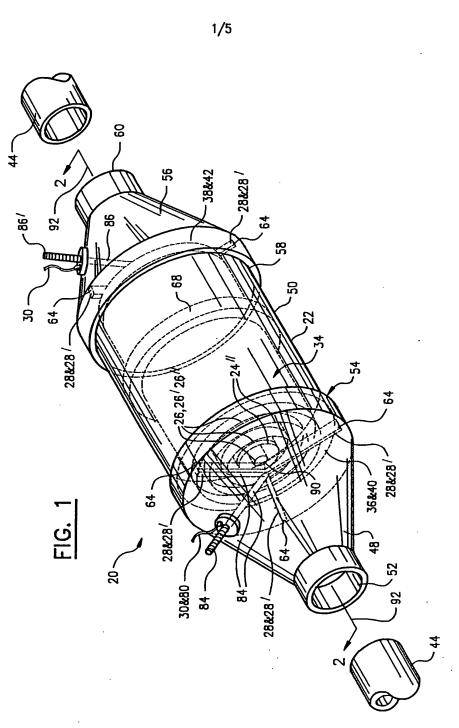
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PCT/US95/01730

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apart water molecules, the direct electrical current further being sufficient to break down the molecular structure of water borne organisms by means of electrolysis and by oxidization created from the breaking apart of the water molecules.

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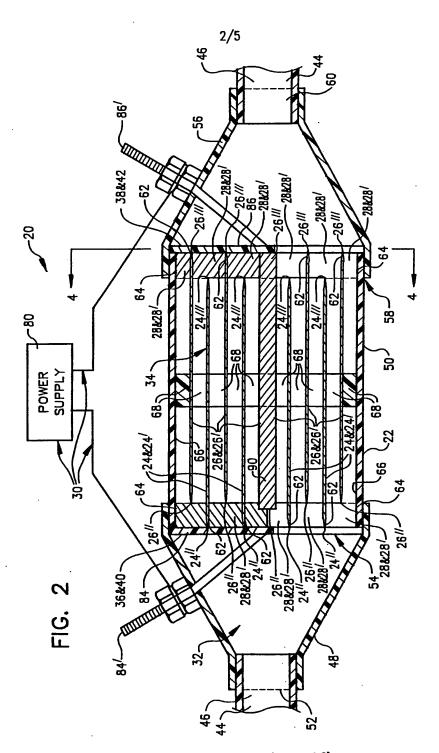
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 244 of 1320

WO 95/21795

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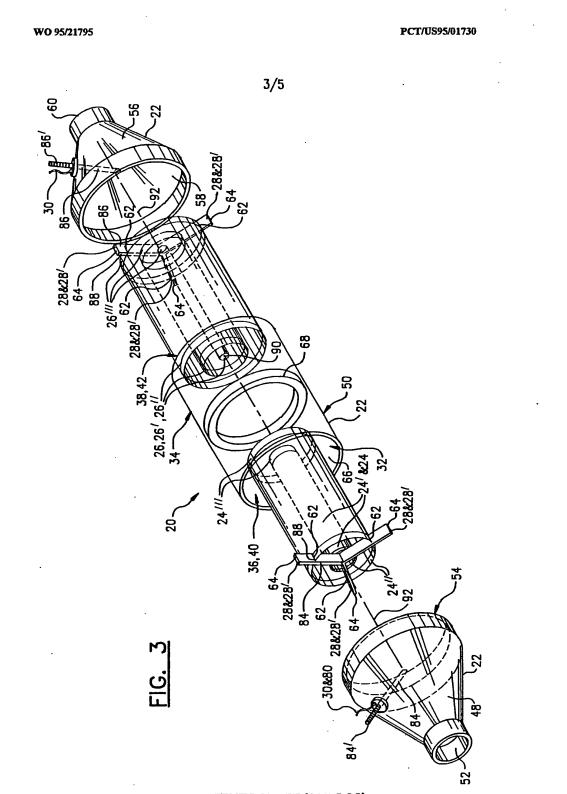
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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 244

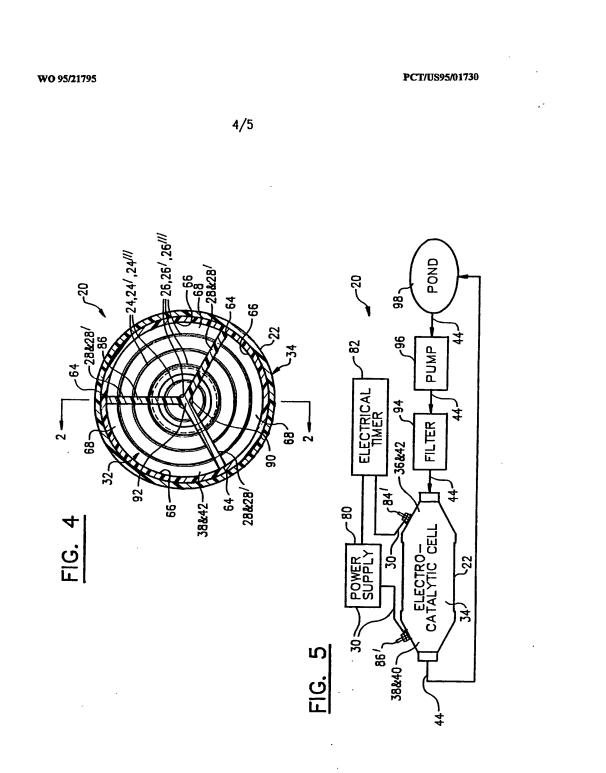
## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 245 of 1320



SUBSTITUTE SHEET (RULE 26)

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 246 of 1320



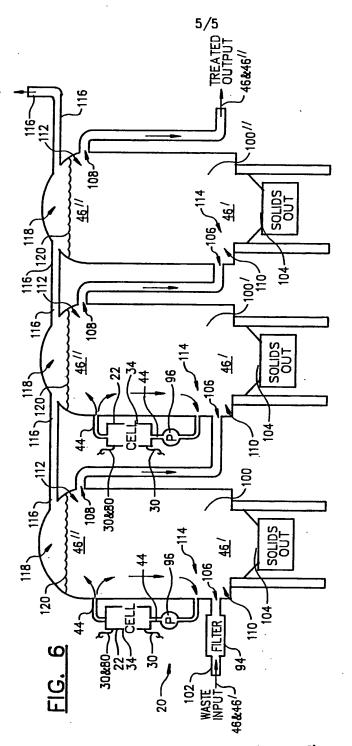
## SUBSTITUTE SHEET (RULE 26)

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 247 of 1320

### WO 95/21795

PCT/US95/01730



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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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IPC(6) US CL	ASSIFICATION OF SUBJECT MATTER :C02F 1/46 : 204/149, 272, 275-278, 290R, 290F to International Patent Classification (IPC) or to both		and IPC		
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U.S. :			10013)		
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Electronic none	data base consulted during the international search (a	ame of data base and,	where practicable	, search terms used)	
C. DO	CUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where a	ppropriate, of the relev	vant passages	Relevant to claim No	
x	US, A, 2,864,750 (HUGHES, JR ET AL) 16 December 1958, 1, Figures and col. 3, lines 40-57 and col. 5, lines 43-50.			1, 11-15, 24-2	
x	US, A, 3,539,486 (FLECK) 10 November 1970, col. 1, lines 35-60 and col. 3, lines 5-34.			1, 10-13, 22 24-25	
x	US, A, 3,654,119 (WHITE ET AL) 04 April 1972, Figure 1, col. 3, lines 45-55 and col. 5, line 2.			1, 10, 18, 24 25	
x	US, A, 4,572,775 (PANIAGUA) 25 February 1986, Figure 1 and col. 3, lines 48-60.		1, 10-12, 19 24-25		
x	US, A, 4,917,782 (DAVIES) 17 April 1990, Figures, col. 3, 1, 10-12, 18-2 lines 5-24 and 64-66 and col. 5, lines 29-40.				
X       Further documents are listed in the continuation of Box C.       See patent family annex.         *       Special categories of cited documents:       T       Inter document published after the international filing date or priority.					
•A• d	ocument defining the general state of the art which is not considered s be of particular relevance	date and not in principle or th	conflict with the applic cory underlying the inv	ation but cited to understand the ention	
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۳ ۵ °0	ited to establish the publication date of another citation or other pocial reason (as specified) occurrent referring to an oral disclosure, use, exhibition or other	"Y" document of particular relevance; the claimed investion cannot be considered to involve an investive step when the document is combined with one or more other such documents, such combination bine abuint to a more while in the while in the state of			
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Date of the	e actual completion of the international search CH 1995	Date of mailing of th	e international ser 5 MAY 199	•	
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Washingt	m. D.C. 20231 No. (703) 305-3230	Telephone No. (703) 308-3327			

Form PCT/ISA/210 (second sheet)(July 1992)\*

**OWT Ex. 2118** Tennant Company v. OWT IPR2021-00625

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 249 of 1320

INTERNATIONAL SEARCH REPORT International ap PCT/US95/01			
C (Continua	tion). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relev	Relevant to claim No.	
x	US, A, 4,936,979 (BROWN) 26 June 1990, Figure 1 a lines 40-68 and col. 3, lines 1-35.	1, 13, 18-19	
A	US, A, 3,563,879 (RICHARDS ET AL) 16 February Figure 1.	1	
A	US, A, 4,525,272 (HENSON) 25 June 1985, Figure 1	1	
A	US, A, 4,915,846 (THOMAS, JR ET AL) 10 April 19 Figures.	1	
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Form PCT/ISA/210 (continuation of second sheet)(July 1992)\*

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 250 of 1320

L Number	Hits	Search Text	DB	Time stamp
1	3929	electrolysis near5 water	USPAT	2003/09/24 14:09
2	1726	microbubble\$2 or nanobubble\$2	USPAT	2003/09/24 14:54
3	11	(microbubble\$2 or nanobubble\$2) and	USPAT	2003/09/24 14:17
		(electrolysis near5 water)		
4	22385	oxygena\$6	USPAT	2003/09/24 14:18
5	100	oxygena\$6 and (electrolysis near5 water)	USPAT	2003/09/24 14:42
6	9	oxygen adj1 emitter	USPAT	2003/09/24 14:43
7	1	5534143.pn.	USPAT	2003/09/24 14:44
8	1	6394429.pn.	USPAT	2003/09/24 14:45
9	1	5982609.pn.	USPAT	2003/09/24 14:46
10	1	4252856.pn.	USPAT	2003/09/24 14:53
11	452	205/755-758,626,628,633.ccls.	USPAT	2003/09/24 14:53
13	2	205/755-758,626,628,633.ccls. and	USPAT	2003/09/24 14:55
		((microbubble\$2 or nanobubble\$2) near10		
		(oxygen or O.\$2.))		
12	98	(microbubble\$2 or nanobubble\$2) near10	USPAT	2003/09/24 14:56
		(oxygen or 0.\$2.)		

Search History 9/24/03 3:06:16 PM Page 1 C:\APPS\east\workspaces\Bruce's workspace1.wsp

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Attorney Docket AQI001US1

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APR 2 8 2003

## PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

TRADEMIN re Application of: James Andrew Senkiw Serial Number: 10/372,292 Title: MICROBUBBLES OF OXYGEN Filing date: 21 February 2003

Examiner: Not assigned Group art unit: 1764

BOX MISSING PARTS Assistant Commissioner for Patents Washington, D.C. 20231

I am transmitting herewith the attached:

X Copy of Notice to File Missing Parts (2 pages) ★ Check for \$580 for filing fee, surcharge and additional claim fees

**X** Formal drawings (5 pages)

\_\_\_\_ Assignment (1 page)

Check for \$40 to record assignment

 $\mathbf{X}$  Declaration and Power of Attorney (**1** pages)

X Return postcard

Bv Kathleen R. Terry Date

23 (Jpn) 03

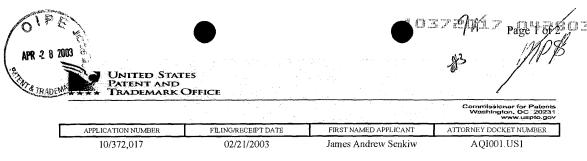
Reg. No. 31884 2417 Como Avenue St. Paul, MN 55108-1459

I hereby certify that these papers are being deposited with the USPS Service with sufficient first class postage and addressed to BOX MISSING PARTS, Commissioner for Patents, Washington D.C. 20231 on the date noted above.



**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 252 of 1320



Kathleen R. Terry 2417 Como Avenue St. Paul, MN 55108-1459 CONFIRMATION NO. 9911 FORMALITIES LETTER

\*0C00000009837851\*

Date Mailed: 04/15/2003

### NOTICE TO FILE MISSING PARTS OF NONPROVISIONAL APPLICATION

FILED UNDER 37 CFR 1.53(b)

#### Filing Date Granted

#### Items Required To Avoid Abandonment:

An application number and filing date have been accorded to this application. The item(s) indicated below, however, are missing. Applicant is given **TWO MONTHS** from the date of this Notice within which to file all required items and pay any fees required below to avoid abandonment. Extensions of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR 1.136(a).

- The statutory basic filing fee is missing.
- Applicant must submit \$ 375 to complete the basic filing fee for a small entity.
- The oath or declaration is unsigned.
- To avoid abandonment, a late filing fee or oath or declaration surcharge as set forth in 37 CFR 1.16(e) of \$65 for a small entity in compliance with 37 CFR 1.27, must be submitted with the missing items identified in this letter.

The application is informal since it does not comply with the regulations for the reason(s) indicated below.

The required item(s) identified below must be timely submitted to avoid abandonment:

- Replacement drawings in compliance with 37 CFR 1.84 and 37 CFR 1.121 are required. The drawings submitted are not acceptable because:
  - The drawings must be reasonably free from erasures and must be free from alterations, overwriting, interlineations, folds, and copy marks. See Figure(s) 3.
  - The drawings have a line quality that is too light to be reproduced (weight of all lines and letters must be heavy enough to permit adequate reproduction) or text that is illegible (reference characters, sheet numbers, and view numbers must be plain and legible) see 37 CFR 1.84(l) and (p)(1)); See Figure(s) 1.

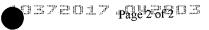
#### Items Required To Avoid Processing Delays:

The item(s) indicated below are also required and should be submitted with any reply to this notice to avoid further processing delays.



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 Additional claim fees of \$140 as a small entity, including any required multiple dependent claim fee, are required. Applicant must submit the additional claim fees or cancel the additional claims for which fees are due.

#### SUMMARY OF FEES DUE:

Total additional fee(s) required for this application is \$580 for a Small Entity

- \$375 Statutory basic filing fee.
- \$65 Late oath or declaration Surcharge.
- Total additional claim fee(s) for this application is \$140
  - \$140 for multiple dependent claim surcharge.

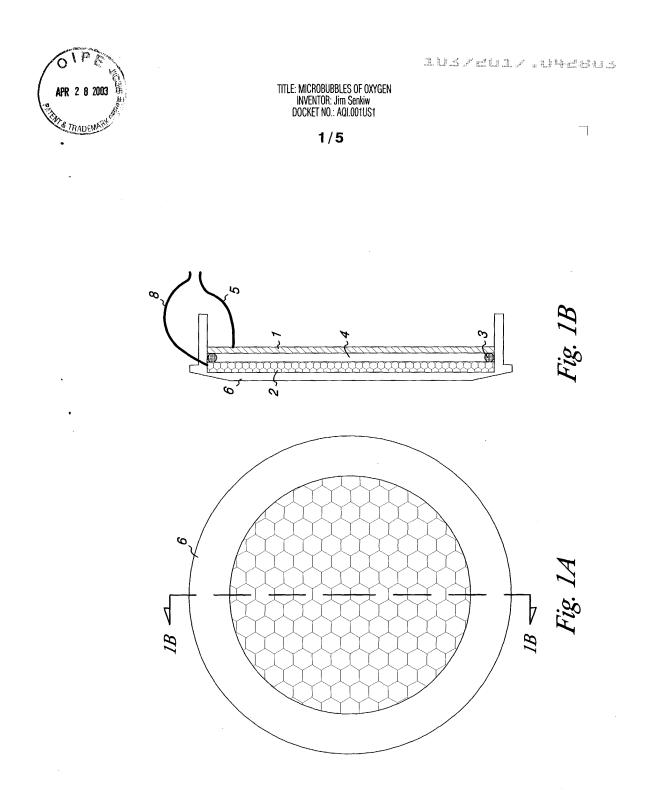
A copy of this notice <u>MUST</u> be returned with the reply.

omer Service Center

Initial Patent Examination Division (703) 308-1202 PART 2 - COPY TO BE RETURNED WITH RESPONSE



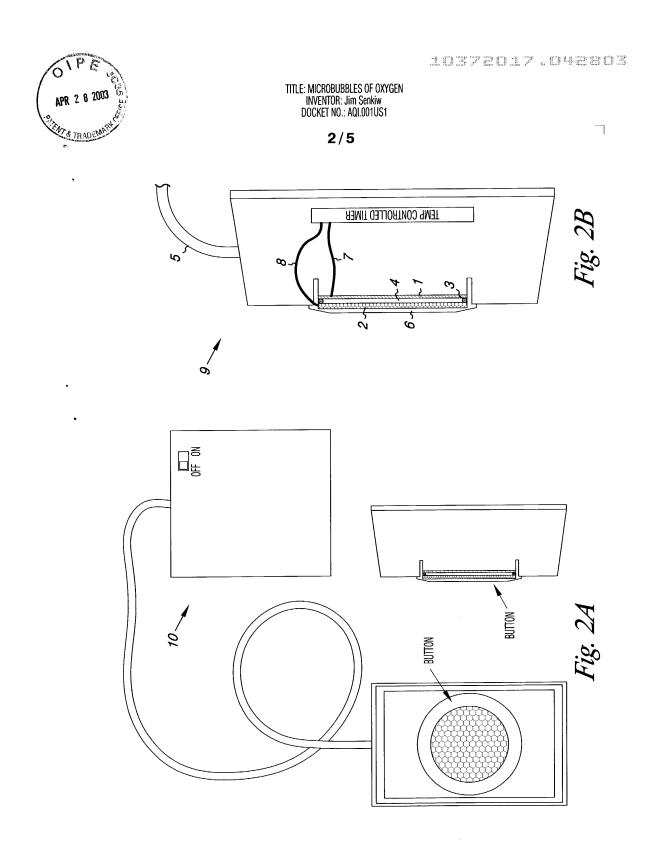
# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 254 of 1320



OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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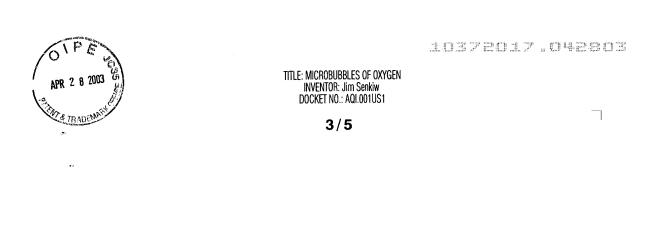
# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 255 of 1320



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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 256 of 1320



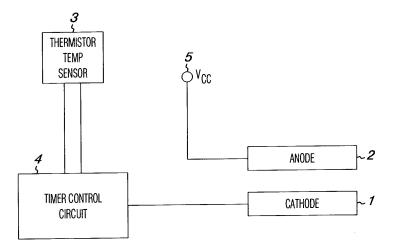


Fig. 3

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 257 of 1320

10372017.042803 1P TITLE: MICROBUBBLES OF OXYGEN INVENTOR: Jim Senkiw DOCKET NO.: AQI.001US1 APR 2 8 2003 4/5 TRADEMP Fig. 4  $\dot{\sigma}$ 

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

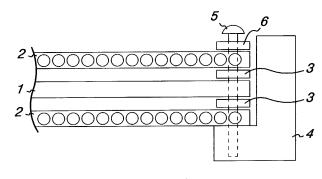
# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 258 of 1320



10372017.042903

TITLE: MICROBUBBLES OF OXYGEN INVENTOR: Jim Senkiw DOCKET NO.: AQI.001US1







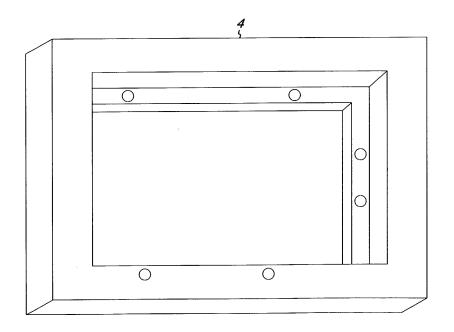


Fig. 5B

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## United States Patent Application COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled: <u>MICROBUBBLES OF OXYGEN</u>

The specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with 37 C.F.R. § 1.56 (attached hereto). I also acknowledge my duty to disclose all information known to be material to patentability which became available between a filing date of a prior application and the national or PCT international filing date in the event this is a Continuation-In-Part application in accordance with 37 C.F.R. §1.63(e).

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed:

#### No such claim for priority is being made at this time.

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

Application Number	Filing Date
60/358,534	22 February 2002

I hereby claim the benefit under 35 U.S.C. § 120 or 365(c) of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose material information as defined in 37 C.F.R. § 1.56(a) which became available between the filing date of the prior application and the national or PCT international filing date of this application:

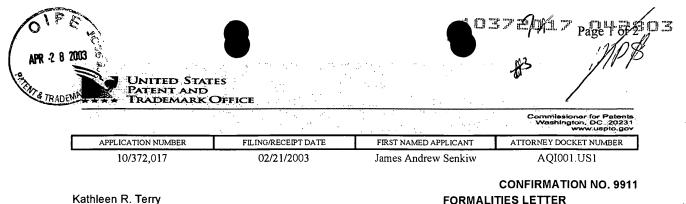
No such claim for priority is being made at this time.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 260 of 1320

10372017.042903

Attorney Docket No.:AQI001.US1 Serial No. not assigned Filing Date: not assigned	Page 3 of 4
I hereby appoint the following attorney(s) and/or patent age all business in the Patent and Trademark Office connected herewith	ent(s) to prosecute this application and to transact
Terry, Kathleen R. Reg. No. 31,884	
I hereby authorize them to act and rely on instructions from and commu firm/organization/who/which first sends/sent this case to them and by whom/whi to be represented unless/until I instruct Kathleen R. Terry to the contrary.	nicate directly with the person/assignee/attorney/ ch I hereby declare that I have consented after full disclosure
Please direct all correspondence in this case to Kathleen R. Terry at the address 2417 Como Avenue, St. Paul, Minn Telephone No. (651) 659	esota 55108-1459
I hereby declare that all statements made herein of my own knowledge belief are believed to be true; and further that these statements were made with the made are punishable by fine or imprisonment, or both, under Section 1001 of Tit statements may jeopardize the validity of the application or any patent issued the	he knowledge that willful false statements and the like so le 18 of the United States Code and that such willful false
Full Name of joint inventor number       1 : James Andrew Senkiw         Citizenship:       United States of America       Reside         Post Office Address:       4750 Aldrich Avenue North       Minneapolis, MN 5430/4529	lence: Minneapolis, MN
Signature: James Andrew Senkiw	Date: 23 Apr 2003
Full Name of joint inventor number 2 : Citizenship: Residence: Post Office Address:	
Signature:	Date:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625



Kathleen R. Terry 2417 Como Avenue St. Paul, MN 55108-1459

Date Mailed: 04/15/2003

\*OC00000009837851\*

## NOTICE TO FILE MISSING PARTS OF NONPROVISIONAL APPLICATION

#### FILED UNDER 37 CFR 1.53(b)

#### Filing Date Granted

## Items Required To Avoid Abandonment:

An application number and filing date have been accorded to this application. The item(s) indicated below, however, are missing. Applicant is given **TWO MONTHS** from the date of this Notice within which to file all required items and pay any fees required below to avoid abandonment. Extensions of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR 1.136(a).

• The statutory basic filing fee is missing.

Applicant must submit \$ 375 to complete the basic filing fee for a small entity.

- The oath or declaration is unsigned.
- To avoid abandonment, a late filing fee or oath or declaration surcharge as set forth in 37 CFR 1.16(e) of \$65 for a small entity in compliance with 37 CFR 1.27, must be submitted with the missing items identified in this letter.

The application is informal since it does not comply with the regulations for the reason(s) indicated below.

The required item(s) identified below must be timely submitted to avoid abandonment:

- Replacement drawings in compliance with 37 CFR 1.84 and 37 CFR 1.121 are required. The drawings submitted are not acceptable because:
  - The drawings must be reasonably free from erasures and must be free from alterations, overwriting, interlineations, folds, and copy marks. See Figure(s) 3.
  - The drawings have a line quality that is too light to be reproduced (weight of all lines and letters must be heavy enough to permit adequate reproduction) or text that is illegible (reference characters, sheet numbers, and view numbers must be plain and legible) see 37 CFR 1.84(l) and (p)(1)); See Figure(s) 1.

#### Items Required To Avoid Processing Delays:

The item(s) indicated below are also required and should be submitted with any reply to this notice to avoid further processing delays.

**JA259** 

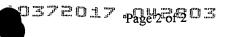


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375.00 65.00 140.00

Page 261





• Additional claim fees of **\$140** as a small entity, including any required multiple dependent claim fee, are required. Applicant must submit the additional claim fees or cancel the additional claims for which fees are due.

#### SUMMARY OF FEES DUE:

Total additional fee(s) required for this application is \$580 for a Small Entity

- \$375 Statutory basic filing fee.
- \$65 Late oath or declaration Surcharge.
- Total additional claim fee(s) for this application is \$140
  - \$140 for multiple dependent claim surcharge.

A copy of this notice <u>MUST</u> be returned with the reply.

Customer Service Center Initial Patent Examination Division (703) 308-1202 PART 2 - COPY TO BE RETURNED WITH RESPONSE

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 263 of 1320

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Attorney Docket AOI001/US1

APR 2 8 2003

PATÉNT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

TADEMATIN re Application of: James Andrew Senkiw Serial Number: 10/372,292 Title: MICROBUBBLES OF OXYGEN Filing date: 21 February 2003

Examiner: Not assigned Group art unit: 1764

BOX MISSING PARTS Assistant Commissioner for Patents Washington, D.C. 20231

I am transmitting herewith the attached:

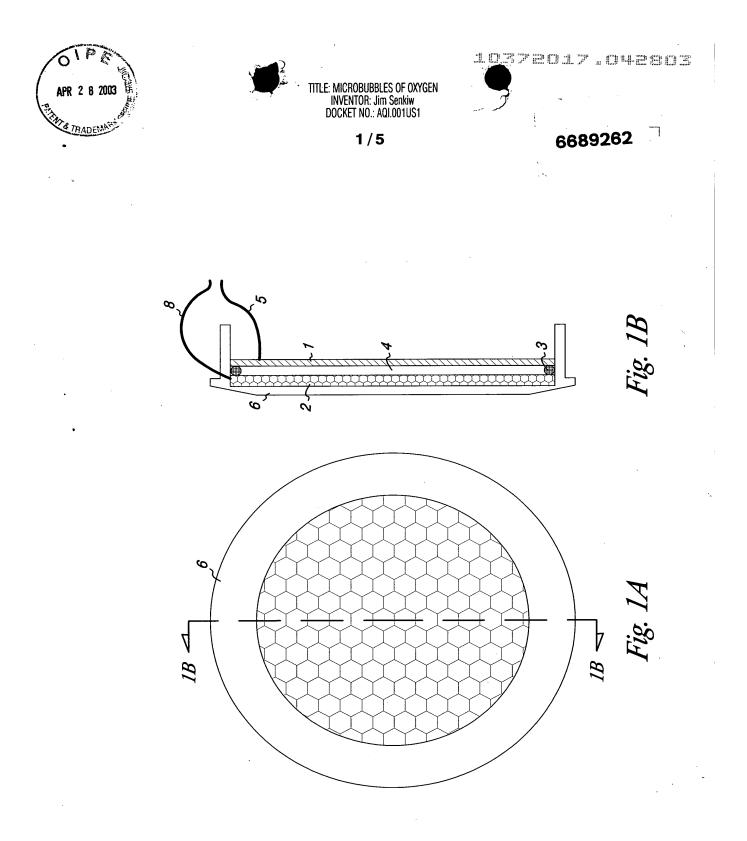
- Copy of Notice to File Missing Parts (2 pages)
- X Check for \$580 for filing fee, surcharge and additional claim fees
- $\mathbf{X}$  Formal drawings (5 pages)
- \_\_\_\_ Assignment (1 page)
- Check for \$40 to record assignment
- Z Declaration and Power of Attorney (2 page)
- X Return postcard

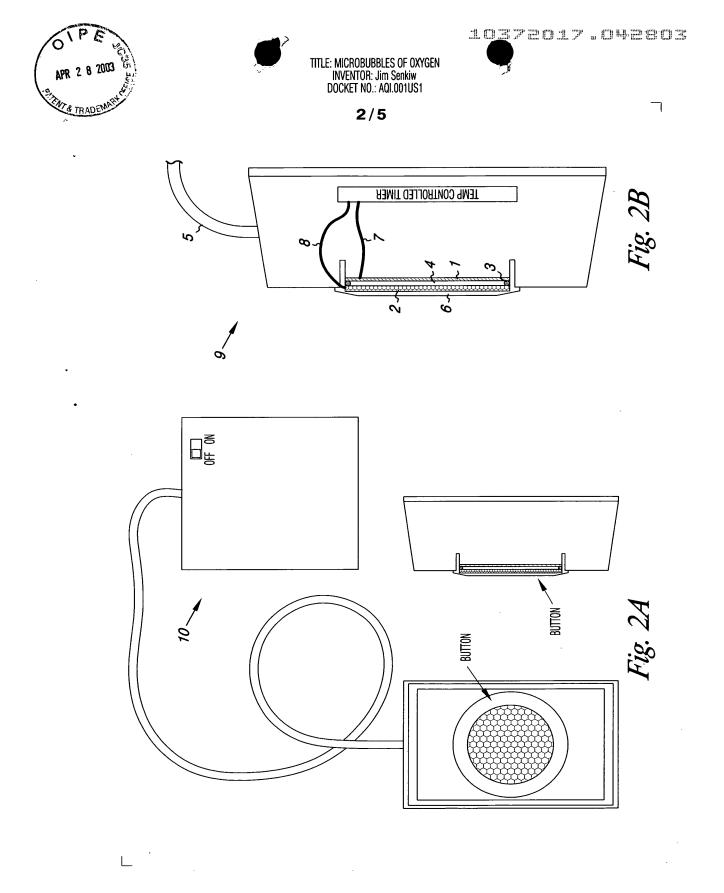
By: Kathleen R. Terry

Reg. No. 31884 2417 Como Avenue St. Paul, MN 55108-1459

23 ( Date

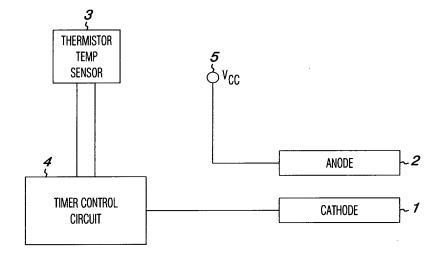
I hereby certify that these papers are being deposited with the USPS Service with sufficient first class postage and addressed to BOX MISSING PARTS, Commissioner for Patents, Washington D.C. 20231 on the date noted above.





CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 266 of 1320



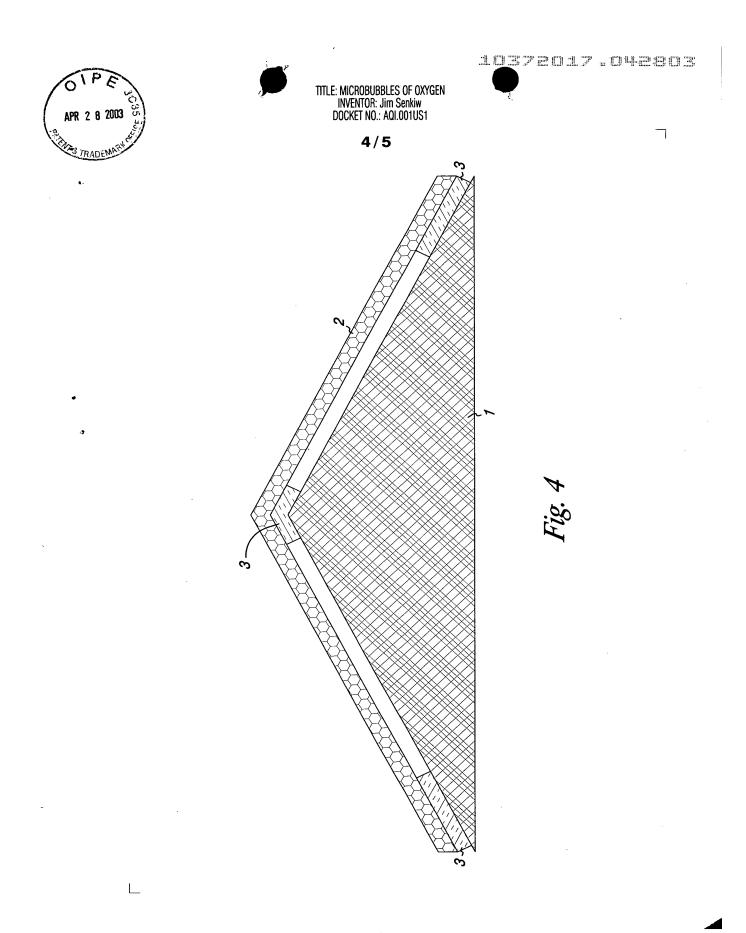


*Fig. 3* 

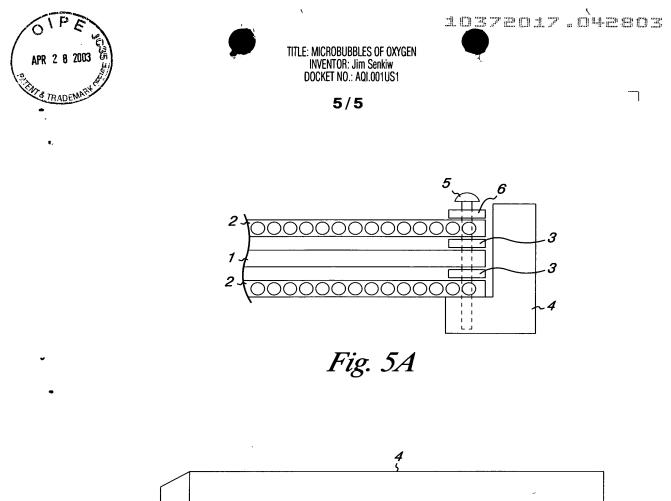
OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 267 of 1320



CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 268 of 1320



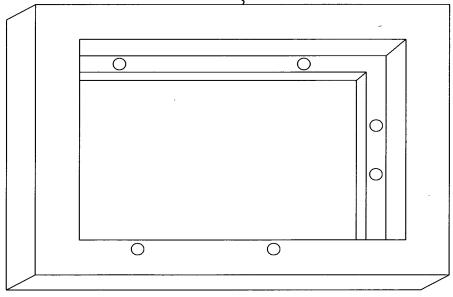


Fig. 5B

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625



COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled: <u>MICROBUBBLES OF OXYGEN</u>

The specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with 37 C.F.R. § 1.56 (attached hereto). I also acknowledge my duty to disclose all information known to be material to patentability which became available between a filing date of a prior application and the national or PCT international filing date in the event this is a Continuation-In-Part application in accordance with 37 C.F.R. §1.63(e).

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed:

## No such claim for priority is being made at this time.

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

# Application NumberFiling Date60/358,53422 February 2002

I hereby claim the benefit under 35 U.S.C. § 120 or 365(c) of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose material information as defined in 37 C.F.R. § 1.56(a) which became available between the filing date of the prior application and the national or PCT international filing date of this application:

## No such claim for priority is being made at this time.

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 270 of 1320

Attorney Docket No.:AQI001.US1 Serial No. not assigned Filing Date: not assigned	 Page 3 of 4

I hereby appoint the following attorney(s) and/or patent agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith:

Terry, Kathleen R. Reg. No. 31,884

I hereby authorize them to act and rely on instructions from and communicate directly with the person/assignee/attorney/ firm/organization/who/which first sends/sent this case to them and by whom/which I hereby declare that I have consented after full disclosure to be represented unless/until I instruct Kathleen R. Terry to the contrary.

Please direct all correspondence in this case to Kathleen R. Terry at the address indicated below: 2417 Como Avenue, St. Paul, Minnesota 55108-1459 Telephone No. (651) 659-9819

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full Name of joint inventor num	ber 1 : James Andrew Senkiw			
Citizenship:	United States of America	Residence: Minne	eapolis, MN	
Post Office Address:	4750 Aldrich Avenue North			
Signature: Jame	Minneapolis, MN 54304529	Date:	23 Apr 2003	 
Full Name of joint inventor num Citizenship: Post Office Address:	ber 2 : Residence	:e:		

Signature:

Date:

			Page 1 of 2
UNITED STAT PATENT AND TRADEMARK			· · · ·
· · · · · · · · · · · · · · · · · · ·			Commissioner for Patents Washington, DC::20231 www.uspto.gov
APPLICATION NUMBER	FILING/RECEIPT DATE	FIRST NAMED APPLICANT	ATTORNEY DOCKET NUMBER
10/372,017	02/21/2003	James Andrew Senkiw	AQI001.US1
Kathleen R. Terry			CONFIRMATION NO. 9911

Kathleen R. Terry 2417 Como Avenue St. Paul, MN 55108-1459

# Date Mailed: 04/15/2003

·OC00000009837851\*

## NOTICE TO FILE MISSING PARTS OF NONPROVISIONAL APPLICATION

## FILED UNDER 37 CFR 1.53(b)

## Filing Date Granted

#### Items Required To Avoid Abandonment:

An application number and filing date have been accorded to this application. The item(s) indicated below, however, are missing. Applicant is given **TWO MONTHS** from the date of this Notice within which to file all required items and pay any fees required below to avoid abandonment. Extensions of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR 1.136(a).

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- Applicant must submit \$ 375 to complete the basic filing fee for a small entity.
- The oath or declaration is unsigned.
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#### Items Required To Avoid Processing Delays:

The item(s) indicated below are also required and should be submitted with any reply to this notice to avoid further processing delays.

JA269





Page 2 of 2

• Additional claim fees of **\$140** as a small entity, including any required multiple dependent claim fee, are required. Applicant must submit the additional claim fees or cancel the additional claims for which fees are due.

#### SUMMARY OF FEES DUE:

Total additional fee(s) required for this application is \$580 for a Small Entity

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- \$65 Late oath or declaration Surcharge.
- Total additional claim fee(s) for this application is \$140
  - \$140 for multiple dependent claim surcharge.

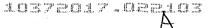
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 273 of 1320

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#### **IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of: James Andrew Senkiw Title: MICROBUBBLES OF OXYGEN Attorney Docket No.: AQI001.US1

## PATENT APPLICATION TRANSMITTAL

#### **BOX PATENT APPLICATION**

Commissioner for Patents Washington, D.C. 20231

We are transmitting herewith the following attached items and information (as indicated with an "X"):

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- X Utility Patent Application under 37 CFR § 1.53(b) comprising:
  - <u>X</u> Specification ( $\underline{13}$  pgs, including claims numbered  $\underline{1}$  through  $\underline{14}$  and a  $\underline{1}$  page Abstract). X Informal Drawing(s) (<u>5</u> sheets).
  - X Unsigned Combined Declaration and Power of Attorney (3 pgs).
- Applicant claims small entity status under 37 C.F.R 1.27. X

	No. Filed	No. Extra	Rate	Fee
TOTAL CLAIMS	14 - 20 =	0	x 9 =	\$0.00
INDEPENDENT CLAIMS	3 - 3 =	0	. x 42 =	\$0.00
[X] MULTIPLE DEPENDENT CLAIMS PRESENTED				\$140.00
BASIC FEE				\$375.00
TOTAL				\$515.00

THE FILING FEE WILL BE PAID UPON RECEIPT OF THE NOTICE TO FILE MISSING PARTS.

Bv: Atty: Kathleen R. Terry Reg. No. 31,884

#### Customer Number 21186

"Express Mail" mailing label number: EU 840 782 877US

Date of Deposit: February 21, 2003

This paper or fee is being deposited on the date indicated above with the United States Postal Service pursuant to 37 CFR 1.10, and is addressed to The Commissioner for Patents, Box Patent Application, Washington, D.C. 20231.



## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 274 of 1320

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## UNITED STATES PATENT APPLICATION

#### MICROBUBBLES OF OXYGEN

Applicant James A. Senkiw 4750 Aldrich Avenue North Minneapolis, MN 55430-3529

United States Citizen

Attorney Docket Number AQI001.US1

Kathleen R. Terry, to whom correspondence should be sent Registration Number 31884 2417 Como Avenue Saint Paul, MN 55108-1459

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 651 659 9819

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## MICROBUBBLES OF OXYGEN

#### FIELD OF THE INVENTION

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This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of aqueous media.

## **BACKGROUND OF THE INVENTION**

10 Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal

- bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic,
- 20 carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. United States Patent Number 5,534,143 discloses a microbubble generator that

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 276 of 1320

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achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. United States Patent Number 6,394,429 discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4 e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O - O_2 + 4H^+ + 4e^-$
NET REACTION:	$6H_2O \rightarrow 4OH^2 + 4H^4 + 2H_2 + O_2$

15 286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic.

20 Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. United States Patent Number 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the

group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 277 of 1320

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according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U. S. Patent Number 4,252,856 comprises vacuum deposited iridium oxide.

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Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

## **RELATED APPLICATIONS**

This application claims priority of United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

## SUMMARY OF THE INVENTION

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This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting 25 of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

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**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 278 of 1320

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

5 Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

10 Controls are provided to regulate the current and timing of electrolysis.

#### **DESCRIPTION OF THE DRAWINGS**

Figure 1 is the  $O_2$  emitter of the invention.

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Figure 2 is an assembled device.

Figure 3 is a diagram of the electronic controls of the  $O_2$  emitter.

Figure 4 shows a funnel or pyramid variation of the  $O_2$  emitter.

Figure 5 shows a multilayer sandwich O<sub>2</sub> emitter.

#### **DETAILED DESCRIPTION OF THE INVENTION**

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Definitions:

For the purpose of describing the present invention, the following terms have these meanings:

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 279 of 1320

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" $O_2$  emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

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The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### **Example 1. Oxygen emitter:**

As shown in Figure 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, OH). The cathode 2 is a 1/16 inch mesh marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. Figure 2 shows a plan view of the assembled device. The O<sub>2</sub> emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon® polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 281 of 1320

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In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. Figure 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### Example 2. Measurement of O<sub>2</sub> bubbles.

Attempts were made to measure the diameter of the  $O_2$  bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exlclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

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Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

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#### Example 3. Other models of oxygen emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. Figure 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

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The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of

models suitable to various uses.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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Emitter Model	Gallons	Volts	Amps Ma	x. Ave	Watts
Bait keeper	. 5	6	0.090	0.060	0.36
Livewell	32	12	0.180	0.120	1.44
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5.000	2.500	30.00

TABLE I

#### Example 4. Multilayer sandwich O<sub>2</sub> emitter

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An  $O_2$  emitter was made in a multilayer sandwich embodiment. (Figure 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

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- cathode screen 0.045 inches thick
- nylon spacer 0.053 inches thick
- anode grid 0.035 inches thick
- nylon spacer 0.053 inches thick
- cathode screen 0.045 inches thick,

for an overall emitter thickness of 0.231 inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 284 of 1320

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of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

JA282

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 285 of 1320

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I claim:

Claim 1. An emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other.

Claim 2. The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support.

Claim 4. The emitter of claim 1 wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 5. The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 6. The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 7. An emitter for electrolytic generation of microbubbles of oxygen comprising a plurality of anodes separated at a critical distance from a plurality of cathodes and a power source all in electrical communication with each other.

Claim 8. A method for keeping aquatic animals emitter alive comprising inserting the emitter of claim 1 or claim 7 into the aquatic medium of the aquatic animals.

Claim 9. The method of claim 8 wherein the aquatic animal is a fish.

Claim 10. The method of claim 8 wherein the aquatic animal is a shrimp.

Claim 11. A method for lowering the biologic oxygen demand of polluted water comprising passing the polluted water through a vessel containing the emitter of claim 1.

Claim 12. The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

Claim 13. An emitter for electrolytic generation of microbubbles of oxygen comprising a platinum-iridium oxide anode on a titanium support separated at a critical distance of from 0.045 inches to 0.060 inches from a stainless steel screen 1/16 inch thick cathode all in electrical communication with a battery.

Claim 14. The emitter of claims 1, 7 or 13 further comprising a timer control.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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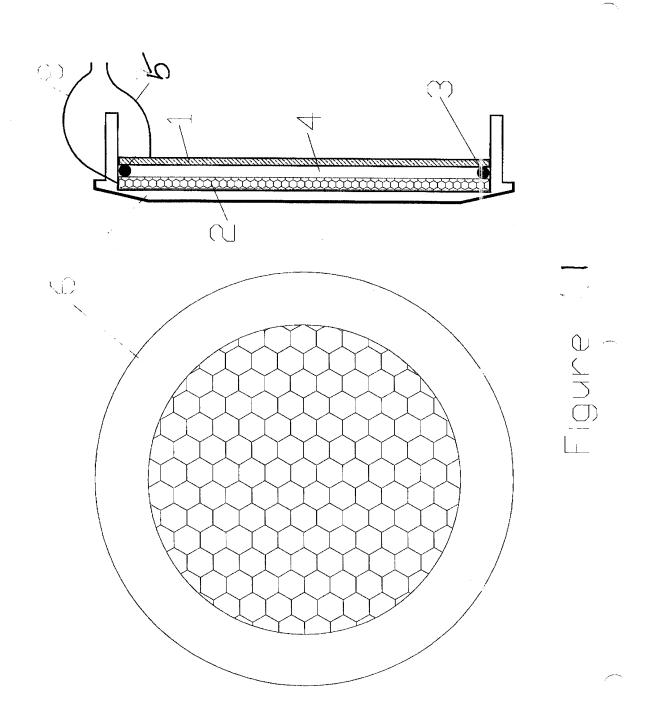
#### ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium. Models suitable for different uses are disclosed.

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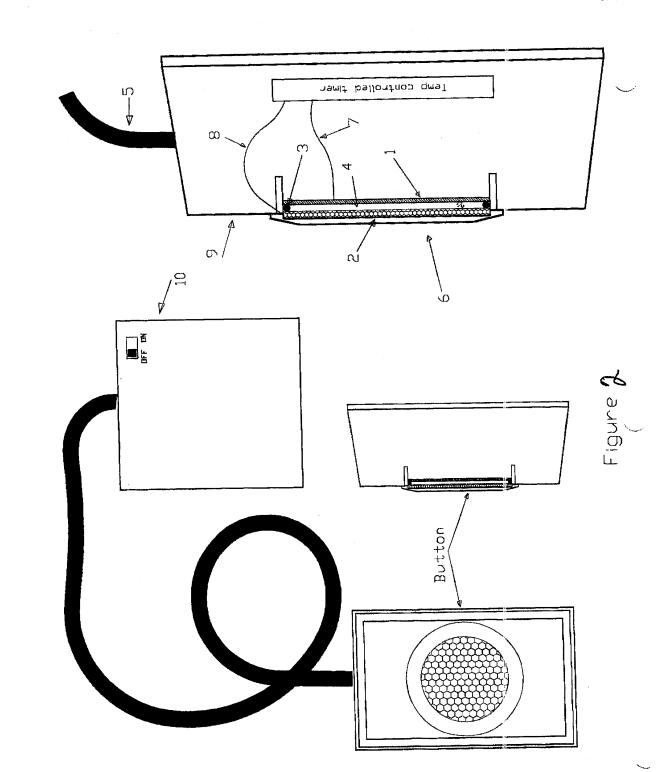
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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 288 of 1320

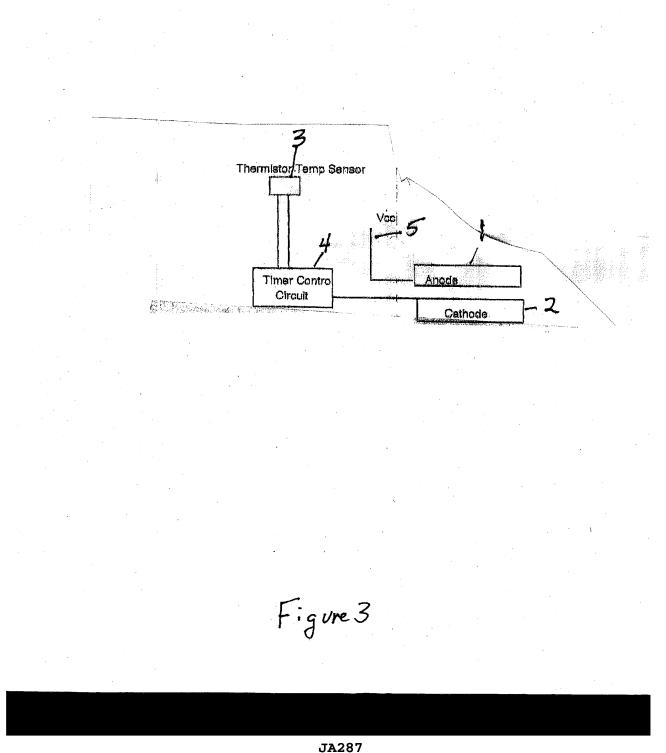
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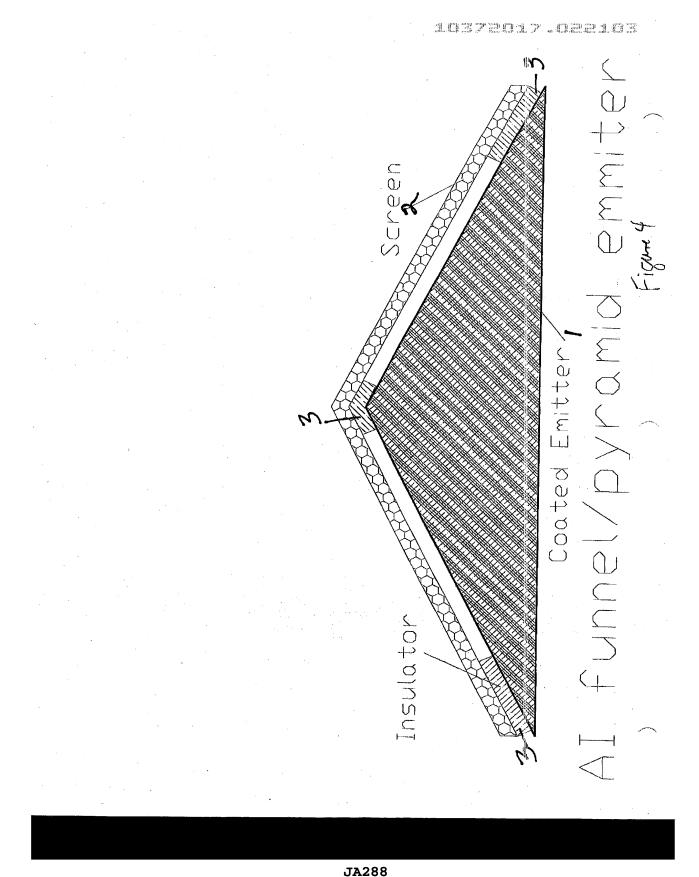
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 289 of 1320

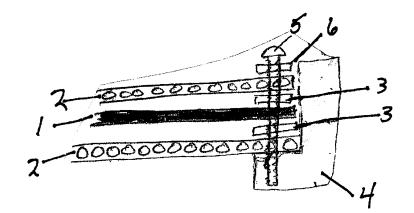
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 290 of 1320



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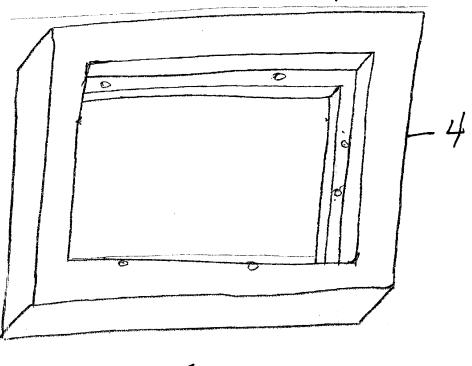


Figure 5

JA289

#### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 292 of 1320

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Attorney Docket No.AQI001.US1

#### United States Patent Application COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled: **MICROBUBBLES OF OXYGEN** 

The specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with 37 C.F.R. § 1.56 (attached hereto). I also acknowledge my duty to disclose all information known to be material to patentability which became available between a filing date of a prior application and the national or PCT international filing date in the event this is a Continuation-In-Part application in accordance with 37 C.F.R. §1.63(e).

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed:

#### No such claim for priority is being made at this time.

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

Application Number	<b>Filing Date</b>
60/358,534	22 February 2002

I hereby claim the benefit under 35 U.S.C. § 120 or 365(c) of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose material information as defined in 37 C.F.R. § 1.56(a) which became available between the filing date of the prior application and the national or PCT international filing date of this application:

No such claim for priority is being made at this time.

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 293 of 1320

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all business in the Patent and Trademark Office connected herewith: Terry, Kathleen R. Reg. No. 31,884 I hereby authorize them to act and rely on instructions from and communicate directly with the person/assignee/attorney/ firm/organization/who/which first sends/sent this case to them and by whom/which I hereby declare that I have consented after full to be represented unless/until I instruct Kathleen R. Terry to the contrary. Please direct all correspondence in this case to <b>Kathleen R. Terry</b> at the address indicated below: 2417 Como Avenue, St. Paul, Minnesota 55108-1459 Telephone No. (651) 659-9819	hereby appoint the following attorney(s) and/or patent agent(s) to prosecute this application and to transact ess in the Patent and Trademark Office connected herewith: en R. Reg. No. 31,884 hereby authorize them to act and rely on instructions from and communicate directly with the person/assignee/attorney/ ization/who/which first sends/sent this case to them and by whom/which I hereby declare that I have consented after full disclose sented unless/until I instruct Kathleen R. Terry to the contrary. tet all correspondence in this case to <b>Kathleen R. Terry</b> at the address indicated below: 2417 Como Avenue, St. Paul, Minnesota 55108-1459 . Telephone No. (651) 659-9819 hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and pelieved to be true; and further that these statements were made with the knowledge that willful false statements and the like so sunishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false may joepardize the validity of the application or any patent issued thereon. of joint inventor number 1 : James Andrew Senkiw p: United States of America e Address: 4750 Aldrich Avenue North Minneapolis, MN 55430-3529 Date: p: Residence: e Address: e Address:	erial No. not assigned					
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#### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 294 of 1320

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Attorney Docket No.:AQ1001.US1 Serial No. not assigned Filing Date: not assigned

Page 3 of 4

§ 1.56 Duty to disclose information material to patentability.

(a) A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is canceled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information which is not material to the patentability of any existing claim. The duty to any existing claim to patentability of any elaim issued in a patent was cited by the Office or submitted to the Office in the manner prescribed by §§ 1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

- (1) prior art cited in search reports of a foreign patent office in a counterpart application, and
- (2) the closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

(b) Under this section, information is material to patentability when it is not cumulative to information already of record or being made of record in the application, and

(1) It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim; or

(2) It refutes, or is inconsistent with, a position the applicant takes in:

(i) Opposing an argument of unpatentability relied on by the Office, or

(ii) Asserting an argument of patentability.

A prima facie case of unpatentability is established when the information compels a conclusion that a claim is unpatentable under the preponderance of evidence, burden-of-proof standard, giving each term in the claim its broadest reasonable construction consistent with the specification, and before any consideration is given to evidence which may be submitted in an attempt to establish a contrary conclusion of patentability.

(c) Individuals associated with the filing or prosecution of a patent application within the meaning of this section are:

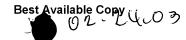
- (1) Each inventor named in the application:
- (2) Each attorney or agent who prepares or prosecutes the application; and
- (3) Every other person who is substantively involved in the preparation or prosecution of the application and who is associated with the inventor, with the assignce or with anyone to whom there is an obligation to assign the application.

(d) Individuals other than the attorney, agent or inventor may comply with this section by disclosing information to the attorney, agent, or inventor.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA292







IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of: James Andrew Senkiw Title: MICROBUBBLES OF OXYGEN Attorney Docket No.: AQI001.US1

### PATENT APPLICATION TRANSMITTAL

#### **BOX PATENT APPLICATION**

Commissioner for Patents Washington, D.C. 20231

We are transmitting herewith the following attached items and information (as indicated with an "X"):

 $\frac{X}{X}$ Return postcard.

....

- Utility Patent Application under 37 CFR § 1.53(b) comprising:
- Specification (<u>1</u>2 pgs, including claims numbered <u>1</u> through <u>14</u> and a <u>1</u> page Abstract). <u>X</u>
- X Informal Drawing(s) (<u>5</u> sheets).
- Unsigned Combined Declaration and Power of Attorney (3 pgs). X
- Applicant claims small entity status under 37 C.F.R 1.27. X

The filing fee (NOT ENCLOSED	) will be calculated as follows:
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	No. Filed	No. Extra	Rate	Fee
TOTAL CLAIMS	14 - 20 =	0 -	x 9 =	\$0.00
INDEPENDENT CLAIMS	3 - 3 =	0	x 42 =	\$0.00
[X] MULTIPLE DEPENDENT CLAIMS PR	\$140.00			
BASIC FEE				\$375.00
	TOTAL			\$515.00

#### THE FILING FEE WILL BE PAID UPON RECEIPT OF THE NOTICE TO FILE MISSING PARTS.

Atty: Kathleen R. Terry Reg. No. 31,884

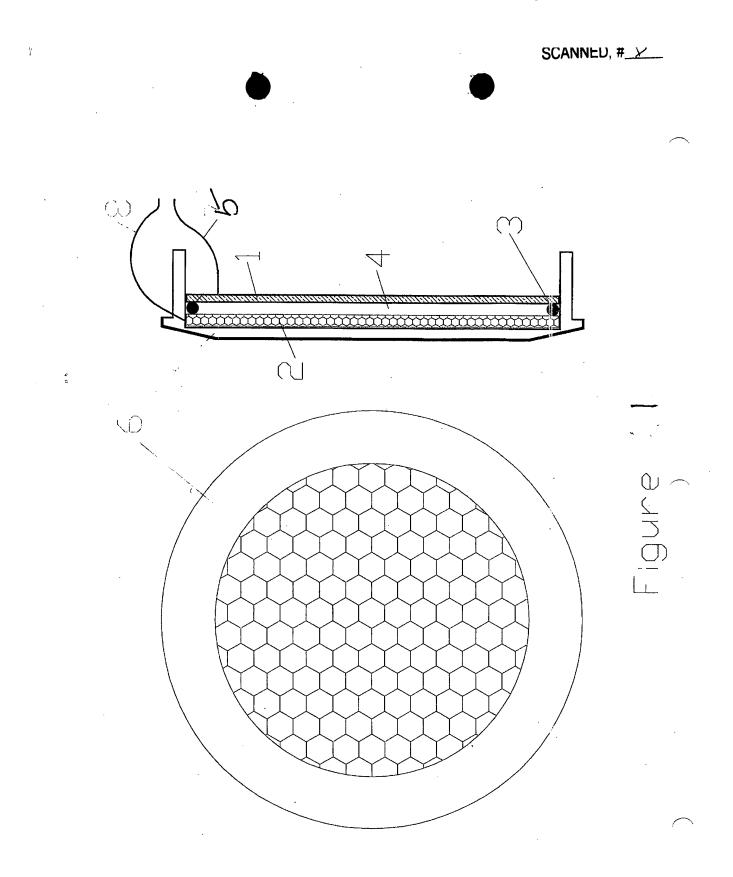
#### Customer Number 21186

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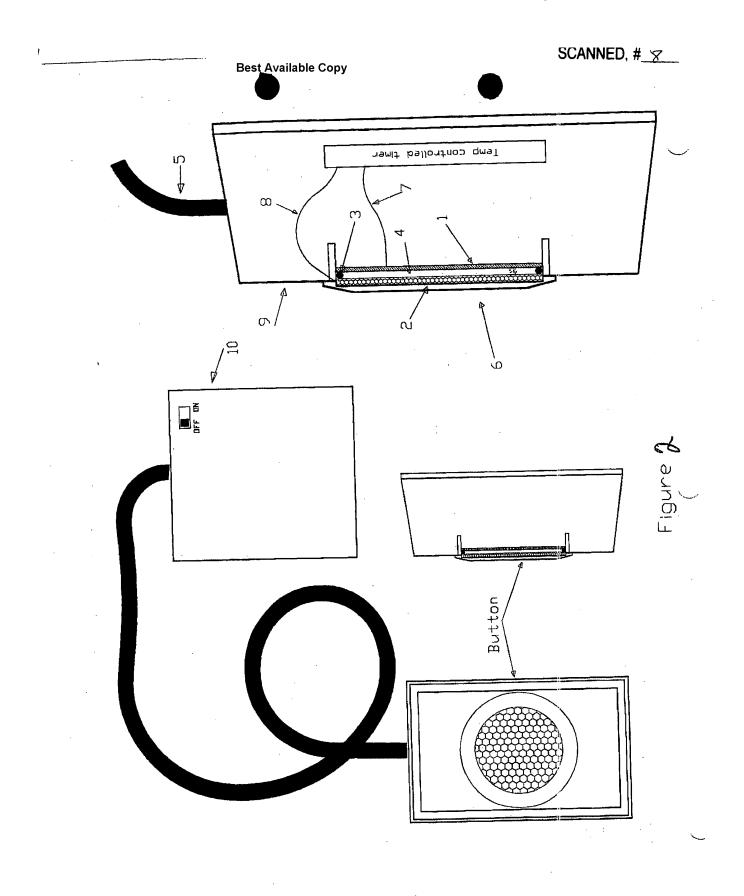
Date of Deposit: February 21, 2003

This paper or fee is being deposited on the date indicated above with the United States Postal Service pursuant to 37 CFR 1.10, and is addressed to The Commissioner for Patents, Box Patent Application, Washington, D.C. 20231.

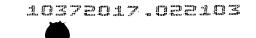
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 297 of 1320







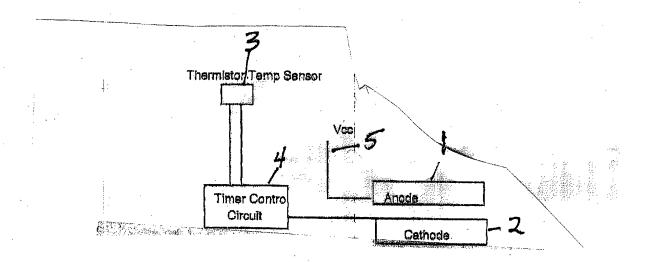
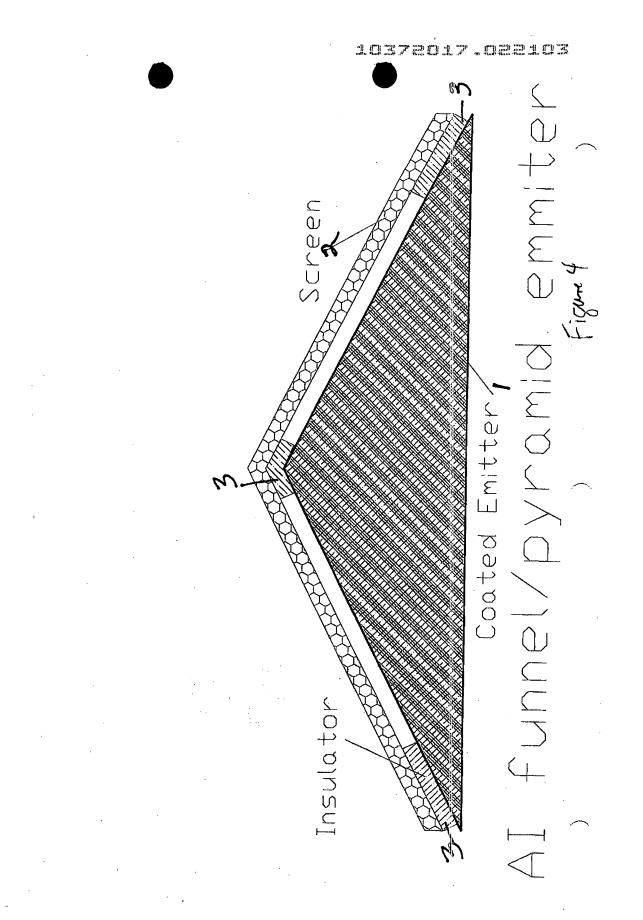


Figure 3

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 300 of 1320

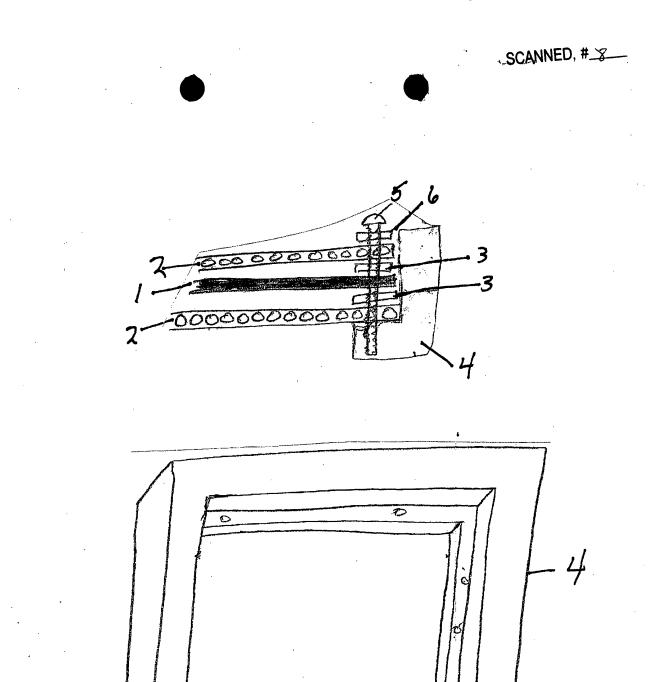


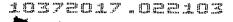
Figure 5

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA298

CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 301 of 1320





## **UNITED STATES PATENT APPLICATION**

#### **MICROBUBBLES OF OXÝGEN**

Applicant James A. Senkiw 4750 Aldrich Avenue North Minneapolis, MN 55430-3529

United States Citizen

Attorney Docket Number AQI001.US1

Kathleen R. Terry, to whom correspondence should be sent Registration Number 31884 2417 Como Avenue Saint Paul, MN 55108-1459

Phone: 651 659 9819 FAX: 651 603 1809 Email: krterry@visi.com

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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#### MICROBUBBLES OF OXYGEN

**FIELD OF THE INVENTION** 

This application cloires the benefit of provisional application (w)/358, 534 fifee 02-22-2002. This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of aqueous media.

#### **BACKGROUND OF THE INVENTION**

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts 10 have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait

- bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal 15 respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic,
- 20 carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

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The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. United States Patent Number 5,534,143 discloses a microbubble generator that

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achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. United States Patent Number 6,394,429 discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4 e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$
NET REACTION:	$6H_2O \rightarrow 4OH^- + 4H^+ + 2H_2 + O_2$

15 286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic.

20 Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. United States Patent Number 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

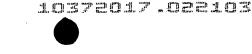
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according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U. S. Patent Number 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the 5 available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping 10 bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

#### **RELATED APPLICATIONS**

This application claims priority of United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

#### SUMMARY OF THE INVENTION

- This invention provides an oxygen emitter which is an electrolytic cell which generates very 20 small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.
- 25 The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

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**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

**JA302** 

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In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

5 Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

10 Controls are provided to regulate the current and timing of electrolysis.

#### **DESCRIPTION OF THE DRAWINGS**

Figure 1 is the  $O_2$  emitter of the invention.

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Figure 2 is an assembled device.

Figure 3 is a diagram of the electronic controls of the  $O_2$  emitter.

20 Figure 4 shows a funnel or pyramid variation of the O<sub>2</sub> emitter.

Figure 5 shows a multilayer sandwich O<sub>2</sub> emitter.

oxygen forms microbubbles and nanobubbles.

#### **DETAILED DESCRIPTION OF THE INVENTION**

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Definitions:

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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"O<sub>2</sub> emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

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"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

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The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

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The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

#### **Example 1. Oxygen emitter:**

As shown in Figure 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, OH). The cathode 2 is a 1/16 inch mesh marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. Figure 2 shows a plan view of the assembled device. The O<sub>2</sub> emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon® polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

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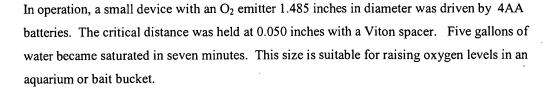
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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. Figure 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### 15 Example 2. Measurement of O<sub>2</sub> bubbles.

Attempts were made to measure the diameter of the  $O_2$  bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exlclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

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Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white

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light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

#### 5 **Example 3.** Other models of oxygen emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. Figure 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

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The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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1.44

1.44

2.16

2.16

3.48

4.92

28.80

30.00



Gallons

5

32

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70

2

50

80

12

12

12

12

12

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Emitter Model

Bait keeper

Livewell

OEM 2 inch

Bait store

Double cycle

OEM 3 inch

OEM 4 inch



TA	BLE I		
Volts	Amps Max.	Ave	Watts
6	0.090	0.060	0.36

0.120

0.120

0.180

0.180

0.265

0.410

0.180

0.210

0.180

0.180

0.500

0.980

Plate 250 12 5.000	2.500

### Example 4. Multilayer sandwich O<sub>2</sub> emitter

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An O<sub>2</sub> emitter was made in a multilayer sandwich embodiment. (Figure 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

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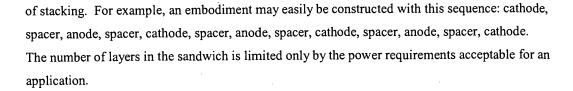
•	cathode screen	0.045 inches thick
•	nylon spacer	0.053 inches thick
•	anode grid	0.035 inches thick
•	nylon spacer	0.053 inches thick
•	cathode screen	0.045 inches thick,
6	<b>.</b>	

for an overall emitter thickness of 0.231 inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence

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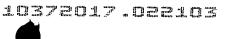


Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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I claim:

Claim 1. An emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other.

Claim 2. The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support.

Claim 4. The emitter of claim 1 wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 5. The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 6. The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim  $\not/$  An emitter for electrolytic generation of microbubbles of oxygen comprising a plurality of anodes separated at a critical distance from a plurality of cathodes and a power source all in electrical communication with each other.

Claim  $\cancel{8}$ . A method for keeping aquatic animals emitter alive comprising inserting the emitter of claim 1 or claim  $\cancel{7}$  into the aquatic medium of the aquatic animals.

Claim  $\frac{9}{2}$  The method of claim 8 wherein the aquatic animal is a fish.

Claim  $\mathcal{W}$ . The method of claim 8 wherein the aquatic animal is a shrimp.

Claim  $\dot{y}$ . A method for lowering the biologic oxygen demand of polluted water comprising passing the polluted water through a vessel containing the emitter of claim 1.

 $\frac{\$}{2}$  Claim  $\frac{1}{2}$ . The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

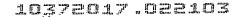
Claim 13. An emitter for electrolytic generation of microbubbles of oxygen comprising a platinum-iridium oxide anode on a titanium support separated at a critical distance of from 0.045 inches to 0.060 inches from a stainless steel screen 1/16 inch thick cathode all in electrical communication with a battery.

Claim 14. The emitter of claims 1, 7 or 13 further comprising a timer control.

11

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA310



#### ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium. Models suitable for different uses are disclosed.

12





Attorney Docket No.AQI001.US1

# United States Patent Application

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled: **MICROBUBBLES OF OXYGEN** 

The specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with 37 C.F.R. § 1.56 (attached hereto). I also acknowledge my duty to disclose all information known to be material to patentability which became available between a filing date of a prior application and the national or PCT international filing date in the event this is a Continuation-In-Part application in accordance with 37 C.F.R. §1.63(e).

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed:

#### No such claim for priority is being made at this time.

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

# Application NumberFiling Date60/358,53422 February 2002

I hereby claim the benefit under 35 U.S.C. § 120 or 365(c) of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose material information as defined in 37 C.F.R. § 1.56(a) which became available between the filing date of the prior application and the national or PCT international filing date of this application:

#### No such claim for priority is being made at this time.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

**JA312** 

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 315 of 1320

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Attorney Docket No.: AQI0 Serial No. not assigned	01.US1			Page 3 of 4
Filing Date: not assigned				
I hereby ar	ppoint the following attorney(s) and/	or patent agent(s) to pr	osecute this applicat	ion and to transact
all business in the	Patent and Trademark Office connect	cted herewith:		
Terry, Kathleen R.	Reg. No. 31,884		•	
firm/organization/wh	norize them to act and rely on instructions fr o/which first sends/sent this case to them an ess/until I instruct Kathleen R. Terry to the	d by whom/which I hereby	tly with the person/assign declare that I have conse	nee/attorney/ ented after full disclosure
Please direct all corre	spondence in this case to Kathleen R. Terr 2417 Como Avenue, Telephon	y at the address indicated b St. Paul, Minnesota 55108 2 No. (651) 659-9819	elow: - <b>1459</b>	
made are punishable statements may jeopa Full Name of joint in Citizenship: Post Office Address:	by fine or imprisonment, or both, under Sec rrdize the validity of the application or any p ventor number 1: <u>James Andrew Sent</u> United States of America 4750 Aldrich Avenue North Minneapolis, MN 55430-3529	atent issued thereon. <u>iw</u> Residence: Mini		
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§ 1.56 Duty to disclose information material to patentability.

(a) A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is canceled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information which is not material to the patentability of any existing claim. The duty to disclose all information which is not material to the patentability of any existing claim. The duty to disclose all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in a patent was cited by the Office or submitted to the Office in the manner prescribed by §§ 1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

- (1) prior art cited in search reports of a foreign patent office in a counterpart application, and
- (2) the closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

(b) Under this section, information is material to patentability when it is not cumulative to information already of record or being made of record in the application, and

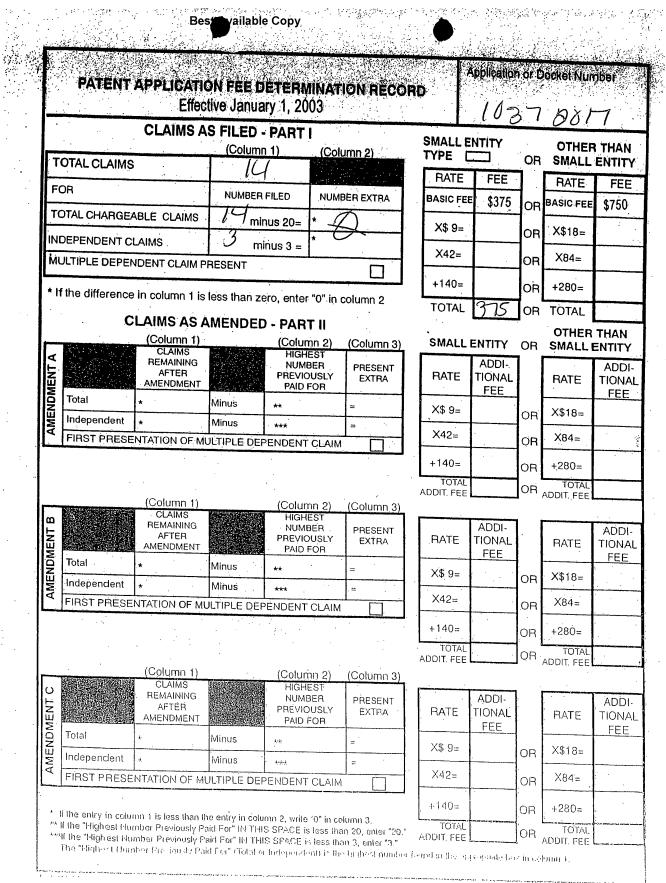
- (1) It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim; or
- (2) It refutes, or is inconsistent with, a position the applicant takes in:
  - (i) Opposing an argument of unpatentability relied on by the Office, or
  - (ii) Asserting an argument of patentability.

A prima facie case of unpatentability is established when the information compels a conclusion that a claim is unpatentable under the preponderance of evidence, burden-of-proof standard, giving each term in the claim its broadest reasonable construction consistent with the specification, and before any consideration is given to evidence which may be submitted in an attempt to establish a contrary conclusion of patentability.

- (c) Individuals associated with the filing or prosecution of a patent application within the meaning of this section are:
  - (1) Each inventor named in the application:
  - (2) Each attorney or agent who prepares or prosecutes the application; and
  - (3) Every other person who is substantively involved in the preparation or prosecution of the application and who is associated with the inventor, with the assignee or with anyone to whom there is an obligation to assign the application.

(d) Individuals other than the attorney, agent or inventor may comply with this section by disclosing information to the attorney, agent, or inventor.

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 317 of 1320



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CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 320 of 1320

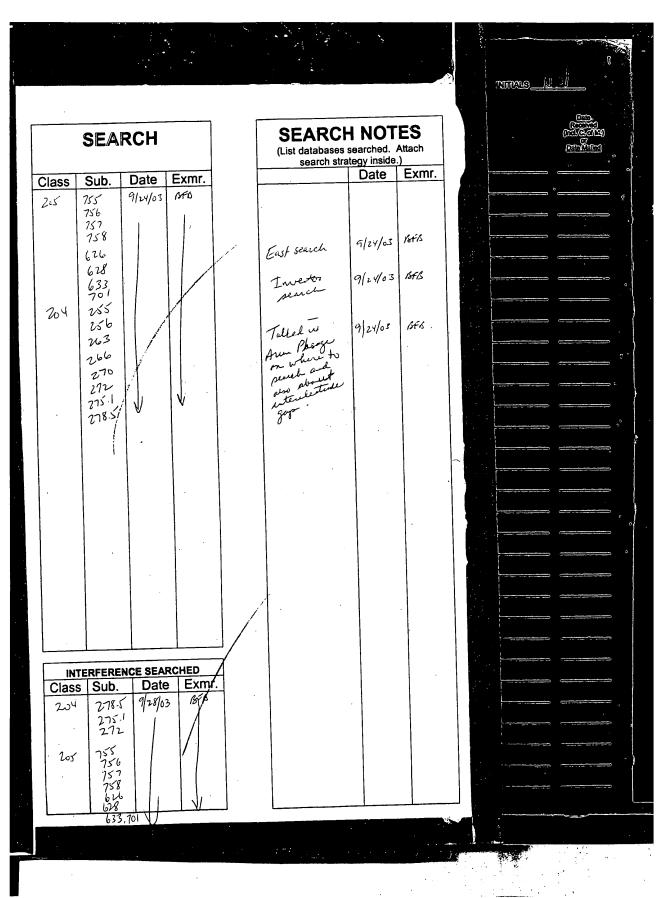
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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 322 of 1320



JA320

## PATENT ASSIGNMENT

## Electronic Version v1.1 Stylesheet Version v1.1

SUBMISSION TYPE	:	NEW ASSIGNMENT										
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		Name	Execution Date									
Oxygenator Water T	Fechnologies, Inc.		10/04/2012									
Aqua Innovations Incorporated 10/04/2012												
RECEIVING PARTY	DATA											
Name:	Roy H Lecy											
Street Address:	2640 North Sau	inders Lake Drive										
City:	Minnetrista											
State/Country:	MINNESOTA											
Postal Code:	55364											
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Patent Number:	70	670495										
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NAME OF SUBMITT	ER:	Nathan M. Brandenburg										

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 324 of 1320

Signature:	/nathanmbrandenburg/
Date:	04/30/2013
	This document serves as an Oath/Declaration (37 CFR 1.63).
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# License Agreement

THIS AGREEMENT ("<u>Agreement</u>") is entered into this 30th day of July, 2008 (the "<u>Effective Date</u>"), by and between Oxygenator Water Technologies, Inc., a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensor</u>") and Aqua Innovations, Inc. a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensee</u>", and Licensor and Licensee each a "<u>Party</u>" and together the "<u>Parties</u>"). Initially capitalized terms defined in this Agreement shall have the meaning ascribed to them respectively herein.

#### WITNESSETH:

LICENSOR owns the technology for which patents have been issued and are pending with respect to electrolytic hydrolysis of water to increase its dissolved oxygen content. A more complete description of said technology, together with a description of the patents issued and currently pending for said technology, is set forth in Article 1 below and in Exhibit "A" attached hereto.

LICENSOR anticipates and intends that it will make additional discoveries and improvements to said technology, some of which may be patentable.

It is further anticipated by the parties that LICENSOR may make improvements to said technology and additional discoveries concerning other applications for said technology.

The parties desire that LICENSOR grant a perpetual, exclusive license to LICENSEE to develop and sell throughout the world certain products utilizing the technology LICENSOR has developed and may in the future develop, all according to the terms and conditions set forth in this Agreement.

The parties further desire that LICENSOR will retain the complete and entire right to develop and sell throughout the world in markets not licensed to LICENSEE hereunder products utilizing the technology LICENSOR has developed and may in the future develop or the technology that LICENSEE may develop in the future, also according to the terms and conditions set forth in this Agreement.

Thus, the parties have agreed to enter into a licensing arrangement by which each party will be entitled to benefit from the other party's patents, technology and know-how concerning electrolytic hydrolysis of water in the sale of products in certain markets.

**NOW, THEREFORE,** based on the foregoing and the mutual covenants and agreements herein contained, the parties hereby covenant and agree as follows:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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38 3<sup>5</sup> - 5 EXHIBIT "B"

# LICENSEE Markets

All worldwide markets for:

- Waste Water Treatment
- Medical Applications
- Sport Fishing
- Aqua Culture
- Horticulture (consumer and commercial)
- Hydroponics

Markets excluded from license agreement (including but not limited to):

- Water Treatment (all applications except waste water)
- Fermentation
- Desalination
- Human Nutrition
- Animal Nutrition

# ARTICLE 1 DEFINITIONS

When used in this Agreement, the following terms have the meanings set forth below unless a different and common meaning of the term is clearly indicated by the context, and variants and derivatives of the following terms shall have correlative meanings:

"Agreement" has the meaning set forth in the preamble.

"LICENSOR Documents" has the meaning set forth in Section 2.6.

"LICENSOR Improvements" means all developments LICENSOR may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable, and which are invented, developed, discovered or otherwise acquired by LICENSOR and which LICENSOR may lawfully communicate to LICENSEE.

"*LICENSOR Markets*" means all uses for the LICENSOR Technology and the LICENSEE Technology other than in the LICENSEE Markets.

"LICENSOR Patents" means all of LICENSOR's patents (whether issued to LICENSOR or controlled by license rights or otherwise and whether such rights are held alone or jointly with others, and patents pending now, or during the term of this Agreement, issued to LICENSOR (by any country) relating to the LICENSOR Technology, including, but not limited to, those patents and those patents pending described on Exhibit A and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSOR Products*" means any product manufactured and/or sold or distributed by LICENSOR or a sub licensee of LICENSOR under any claim contained in the LICENSEE Patents.

"LICENSOR Property" means LICENSOR Patents, LICENSOR Improvements and LICENSOR Technology.

"LICENSOR Technology" means LICENSOR's unpatented technology and information now existing and relating to, and embodying LICENSOR's experience in electrolytic hydrolysis of water. LICENSOR Technology shall include the technical information in all current and future manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSOR to LICENSEE during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSOR imparting the same directly or giving LICENSEE access to any of LICENSOR's production facilities.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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"Effective Date" has the meaning set forth in the preamble.

"LICENSEE Documents" has the meaning set forth in Section 2.7.

"LICENSEE Improvements" means all developments LICENSEE may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable and which are invented, developed, discovered or otherwise acquired by LICENSEE and which LICENSEE may lawfully communicate to LICENSOR.

"*LICENSEE Markets*" means those markets for Licensee Products as are described in Exhibit B attached hereto.

"LICENSEE Patents" means all of LICENSEE's patents (whether issued to LICENSEE or controlled by license rights or otherwise and whether such rights are held alone or jointly with others) which may after the effective date of this Agreement be issued (by any country) relating to electrolytic hydrolysis of water and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSEE Products*" means any product manufactured and/or sold or distributed to any party other than LICENSOR by LICENSEE or a sublicense of LICENSEE in conformity with the terms of this Agreement, including, but not limited to, any product which is based on any claim or thing contained in any LICENSOR Property.

"LICENSEE Property" means LICENSEE Patents, LICENSEE Improvements and LICENSEE Technology.

"LICENSEE Technology" means LICENSEE's unpatented technology and information which LICENSEE may develop relating to, and embodying LICENSEE's experience in, the manufacturing, the processing, quality control, and sale of the LICENSEE Products. LICENSEE Technology shall include the technical information in all manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSEE to LICENSOR during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSEE imparting the same directly or giving LICENSOR access to any of LICENSEE's production facilities.

"Territory" means the world,

# ARTICLE 2 MARKETS AND LICENSING

2.1. Exclusive Markets. The parties agree that unless properly terminated by LICENSOR pursuant to Section 5.1 below, LICENSEE will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSEE Markets in the Territory. The parties further agree that LICENSOR will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSOR Markets in the Territory. LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSOR Products in the LICENSOR Markets in the Territory. LICENSEE may not, directly or indirectly, distribute in any manner any product which competes with the LICENSEE Products in any manner nor may LICENSEE assist or have any interest in any third party distributing any such products through licensing or assignment of technology to any such third party or by any other means.

2.2. Exclusive License to LICENSEE. Subject to the terms and conditions of this Agreement, LICENSOR hereby confers upon LICENSEE the sole and exclusive license, with the right of sublicense, under the LICENSOR Property, to make, have made, use and sell the LICENSEE Products in the LICENSEE Markets in the Territory and to prevent infringement of the LICENSOR Patents, and to prevent unauthorized use and disclosure of the LICENSOR Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSEE Products.

2.3. Exclusive License to LICENSOR. Subject to the terms and conditions of this Agreement, LICENSEE hereby confers upon LICENSOR the sole and exclusive, royalty-free license, with the right of sublicense, under the LICENSEE Property, to make, have made, use and sell the LICENSOR Products in the LICENSOR Markets in the Territory and to prevent infringement of the LICENSEE Patents, and to prevent unauthorized use and disclosure of the LICENSEE Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSOR Products.

**2.4. Product Markings.** The Parties shall insure that all LICENSOR Products and all LICENSEE Products are marked with any applicable patent number and all labeling and other product information shall be marked in such manner as to conform with the patent laws and practices of the country of sale.

2.5 Transfer of Technology by LICENSOR. As promptly as practicable after the execution of this Agreement, LICENSOR shall deliver to LICENSEE all information concerning the LICENSOR Property. LICENSOR also promptly shall deliver to LICENSEE all future information it acquires concerning the LICENSOR Property. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSOR, shall be referred to as "LICENSOR Documents". LICENSEE shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSOR Documents in such manner as to maximize the value of the LICENSOR Patents, the LICENSOR Technology and the LICENSOR Improvements; without limiting the generality of the foregoing, LICENSEE shall, and shall cause its employees and

representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSOR Documents. Upon 10 days advance notice and at reasonable times, LICENSOR shall permit LICENSEE access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSOR to answer fully such questions as LICENSEE may have with a view to transferring the LICENSOR Property. Nothing in this Section 2.6 shall require LICENSOR to disclose to LICENSEE any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

## 2.6. Transfer of Technology by LICENSEE. As promptly as practicable after

LICENSEE develops, discovers or otherwise comes into possession of LICENSEE Patents, LICENSEE Improvements and/or LICENSEE Technology, LICENSEE shall deliver to LICENSOR all information concerning same. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSEE, shall be referred to as "LICENSEE Documents." LICENSOR shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSEE Documents in such manner as to maximize the value of the LICENSEE Patents, the LICENSEE Technology and the LICENSEE Improvements; without limiting the generality of the foregoing, LICENSOR shall, and shall cause its employees and representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSEE Documents. Upon 10 days advance notice and at reasonable times, LICENSEE shall permit LICENSOR access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSEE to answer fully such questions as LICENSOR may have with a view to transferring to LICENSOR the LICENSEE Property. Nothing in this Section 2.7 shall require LICENSEE to disclose to LICENSOR any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

2.7. Further Prosecution of Patents. LICENSOR will continue with the prompt prosecution of all pending patent applications filed by LICENSOR as detailed on Schedule "A", so long as it is commercially reasonable to do so, and LICENSOR will periodically advise LICENSEE of the status of such prosecutions. As soon as practical, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSOR Patents. In the event that LICENSEE files an application for a patent(s) covering electrolytic hydrolysis of water, LICENSEE will periodically advise LICENSOR of the status of the prosecution of any such patent. As soon as practical after any such application by LICENSEE, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSEE Patents. From the date of this Agreement, all expenses incurred in filing for and maintaining protection in those countries mutually agreed upon (other than expenses of prosecuting the original patent application in the first jurisdiction, which will be the responsibility of the Party filing the patent application) will be shared equally by the Parties. Either Party may seek protection in any country not mutually agreed upon by paying the full amount of the cost thereof. A party seeking such additional protection will receive the full cooperation of the other Party (other than in paying the expenses thereof) in protecting all patents in any such other country.

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2.8. Additional Covenants. Each of LICENSOR and LICENSEE shall faithfully comply with their respective obligations under this Agreement and shall incorporate all terms and conditions required by this Agreement in any contracts with third parties to whom access to the LICENSOR Property or the LICENSEE Property, as the case may be, may (but only in accordance with this Agreement) be given. Each of LICENSOR and LICENSEE shall indemnify and hold harmless the other Party and its successors and assigns from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on such Party, and arising out of or in connection with or resulting from the marketing, sale or use of the imdemnifying Party's product(s), including any advertising or other promotional activities related thereto.

**2.9. Infringement Actions.** Neither LICENSOR nor LICENSEE will have any responsibility to the other Party for any damage or expense incurred by such other Party which arises from any action, claim or cause of action brought by any person as the result of any alleged patent infringement or trade secret misappropriation by reason of such other Party's manufacture, use or sale of any product under any of the licenses conferred hereby.

2.10. LICENSEE's Rights in Event of Third Party Infringement. LICENSEE shall have the right, in LICENSOR's name (if required by law, otherwise, in LICENSEE's name) but at LICENSEE's sole expense, to sue third parties in the LICENSEE Markets for infringements of the LICENSOR Patents and misappropriation of the LICENSOR Technology and unpatented LICENSOR Improvements, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. LICENSOR may, if it so elects, join in any such suit as a plaintiff. All damages, awards or settlement proceeds in such suit shall be LICENSEE's. If LICENSEE, after notice from LICENSOR of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSOR, in its own name (or, if required by law, in its and LICENSEE's name) and at its own expense, may sue therefore, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSOR shall promptly reimburse LICENSEE for said suitassociated direct expenses upon presentation of LICENSEE's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's.

2.11. LICENSOR's Rights in Event of Third Party Infringement. LICENSOR

shall have the right, in LICENSEE's name (if required by law, otherwise, in LICENSOR's name) but at LICENSOR's sole expense, to sue third parties in the LICENSOR Markets for infringements of the LICENSEE Patents and misappropriation of the LICENSEE Technology and unpatented LICENSEE Improvements, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSEE may, if it so elects, join in any such suit as a plaintiff. LICENSOR shall promptly reimburse LICENSEE for said suit-associated direct expenses upon presentation of LICENSEE's itemized

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's. If LICENSOR, after notice from LICENSEE of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSEE, in its own name (or, if required by law, in its and LICENSOR's name) and at its own expense, may sue therefore, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSEE's.

**2.12. LICENSEE Royalty Payment.** None. License is granted without cost to LICENSEE.

# ARTICLE 3 INDEMNIFICATION

**3.1.** Indemnification by LICENSEE. LICENSEE shall indemnify and hold LICENSOR and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSOR arising out of or in connection with or resulting from the marketing, sale or use of the LICENSEE Products, including any advertising or other promotional activities related thereto. LICENSOR shall be an added insured party to LICENSEE's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSEE shall procure and have in place no later than the date on which LICENSEE first makes a delivery of any of the LICENSEE Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSOR is provided at least thirty (30) days advance written notice.

**3.2. Indemnification by LICENSOR.** LICENSOR shall indemnify and hold LICENSEE and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSEE arising out of or in connection with or resulting from the marketing, sale or use of the LICENSOR Products, including any advertising or other promotional activities related thereto. At such time, if any, as LICENSOR shall sell LICENSOR Products, LICENSOR shall add LICENSEE as an added insured party to LICENSOR's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSOR shall procure and have in place no later than the date on which LICENSOR first makes a delivery of any of the LICENSOR Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSEE is provided at least thirty (30) days advance written notice.

## ARTICLE 4 CONFIDENTIALITY

4.1. Restrictions on Use and Disclosure of LICENSOR Property by LICENSEE. LICENSEE shall use the LICENSOR Property in confidence and shall not

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate: (a) if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSEE, or (b) upon the expiration of the obligation of LICENSEE under this Agreement to pay royalties to LICENSOR.

4.2. Restrictions on Use and Disclosure of LICENSEE Property by LICENSOR. LICENSOR shall use the LICENSEE Property in confidence and shall not disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSOR.

## 4.3. Employees; Third Parties Etc. In order to faithfully perform their

respective obligations under sections 4.1 and 4.2, the Parties shall limit access to the other Party's Property to only those of its officers, employees and agents who shall have a need to receive or have access to that portion, and then only for the purposes of the practice under the licenses conferred by this Agreement. Each Party will require any third party, to whom access may be authorized under this Agreement, to execute an appropriate confidentiality agreement.

4.4. Authorized Required Disclosures. Nothing in this Article 4 shall prevent a Party: (a) from complying (but only to the narrowest extent required by law and regulation and with due notice on any submissions to governmental agencies of the confidential or proprietary status of the information with a view toward restricting access to, and use or disclosure by, third parties) with reasonable requirements of governmental agencies to disclose information in order to receive legally required consents or permissions to manufacture or sell that Party's Products; or (b) from disclosing information under court order, but only after having made all reasonable efforts to secure the court's order to (i) limit production, use and disclosure practicable under the circumstances and (ii) hold all proceedings in camera with a sealed record.

## ARTICLE 5 RESOLUTION OF DISPUTES

All claims, disputes and other matters in question arising out of, or relating to, this Agreement or the performance thereof shall be submitted to, and determined by, arbitration if good faith negotiations between the parties do not resolve such claim, dispute or other matter within 60 days. Such arbitration shall proceed in accordance with the Commercial Arbitration Rules of the American Arbitration Association then pertaining (the "Rules"), insofar as such Rules are not inconsistent with the provisions expressly set forth in this Agreement, unless the parties mutually agree otherwise, and pursuant to the following procedures:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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(a) Notice of the demand for arbitration shall be filed in writing with the other Member and with the American Arbitration Association. Each Member shall appoint an arbitrator, and those party-appointed arbitrators shall appoint a third neutral arbitrator within 10 days. If the party-appointed arbitrators fail to appoint a third, neutral arbitrator within 10 days, such third, neutral arbitrator shall be appointed by the American Arbitration Association in accordance with the Rules. A determination by a majority of the panel shall be binding.

(b) Reasonable discovery shall be allowed in arbitration.

(c) All proceedings before the arbitrators shall be held in Minneapolis, Minnesota. The governing law shall be as specified in Section 8.1 below.

(d) The costs and fees of the arbitration, including attorneys' fees, shall be allocated by the arbitrators.

(e) The award rendered by the arbitrators shall be final and judgment may be entered in accordance with applicable law and in any court having jurisdiction thereof.

#### ARTICLE 6 NOTICES

**6.1.** Notices. All communications, demands, notices or objections required or permitted to be given or served under this Agreement shall be in writing and shall be deemed to have been duly given or made only if delivered in person, deposited in the United States mail, postage prepaid, for mailing by certified or registered mail, return receipt requested, or delivered by prepaid overnight courier service, addressed to the appropriate party as follows:

- If to LICENSOR: Richard Disrud, COO Aqua Innovations, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435
- If to LICENSEE: Jeffrey Brink, CEO Oxygenator Water Technology, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435

Either party may change its address by giving notice in writing, stating the new address, to the other Party as provided in the foregoing manner. Commencing on the tenth (10th) day after the giving of such notice, such newly designated address shall be such Party's address for the purpose of all communications, demands, notices or objections required or permitted to be given or served under this Agreement.

# ARTICLE 7 MISCELLANEOUS

**7.1. Governing Law; Court Proceedings.** The validity, performance, and all matters relating to the interpretation and effect of this Agreement shall be governed by the internal law in effect in the State of Minnesota without regard to principles of law (such as "conflicts of law") that might make the law of some other jurisdiction applicable. Without limiting the terms set forth in Article 6 with respect to the resolution of disputes, each Party agrees to the exclusive and irrevocable jurisdiction arising out of or in any way related to this Agreement which may be brought in a court of law and both parties agree that personal service from any such court may be effectively served upon a party at the respective addresses set forth in Section 7.1.

**7.2. Exhibits.** Exhibits, schedules and annexes referred to in this Agreement and attached hereto are incorporated herein in full by this reference as if each of such exhibits, schedules or annexes were set forth in the body of this Agreement and duly executed by the parties hereto.

**7.3. Additional Documents and Acts.** Each party agrees that it will use all reasonable efforts to take, or cause to be taken, all actions and to do, or cause to be done, all things necessary, proper or advisable, including, but not limited to, the execution of additional documents and instruments, to consummate, make effective and carry out the transactions contemplated by this Agreement.

7.4. Amendment, Modification or Waiver. No amendment, modification or waiver of any condition, provision or term of this Agreement shall be valid or of any effect unless made in writing, signed by the party or parties to be bound or its duly authorized representative and specifying with particularity the nature and extent of such amendment, modification or waiver. Any waiver by any party of a default of another party shall not affect or impair any right arising from any subsequent default.

**7.5. Severable Provisions.** Whenever possible, each provision of this Agreement will be interpreted in such manner as to be effective and valid under applicable law, but if any provision of this Agreement is held to be invalid, illegal or unenforceable under any applicable law or rule in any jurisdiction, such provision will be ineffective only to the extent of such invalidity, illegality, or unenforceability in such jurisdiction, without invalidating the remainder of this Agreement in such jurisdiction or any provision hereof in any other jurisdiction.

**7.6. Entire Agreement.** This Agreement contains the entire understanding of the parties hereto in respect of the transactions contemplated hereby and supersedes all prior agreements and understandings between the parties with respect to such subject matter.

7.7. Captions, Headings, Titles or References to Gender. All captions, headings

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

or titles in the paragraphs or sections of this Agreement are inserted for convenience of reference only and shall not constitute a part of this Agreement or as a limitation of the scope of the particular paragraphs or sections to which they apply. Where appropriate, the masculine gender may be read as the feminine gender or the neuter gender, the feminine gender may be read as the masculine gender or the neuter gender and the neuter gender may be read as the masculine gender or the feminine gender.

**7.8.** Counterparts. This Agreement may be executed in two (2) or more counterparts, each of which shall be considered one and the same Agreement and shall become effective when one or more counterparts have been signed by each of the parties and delivered to the other parties.

IN WITNESS WHEREOF, the parties have executed this Agreement on the date first written above.

AQUA INNOVATIONS, INC.

1.1.2.=

Dick Disrud its COO

**OXYGENATOR WATER TECHNOLOGIES, INC.** 

Jeff Brink its CEO

EXHIBIT "A"

# DESCRIPTION OF LICENSOR PATENTS AND PATENTS PENDING

## United States Patent Number: US 6,689,262 B2 Date of Patent: February 10, 2004 Name: Microbubbles of Oxygen Application Number: 10/372,017

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen.

United States Patent Number: US 7,396,441 B2 Publication Date: July 8, 2008 Name: Flow-Thru Oxygenator Application Number: 10/732,326

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen. A flow-through model for oxygenating flowing water. The use of supersaturated water for enhancing the growth of plants. Method of applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture. The treatment of waste water by raising the dissolved oxygen with the use of oxygen emitter.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 338 of 1320

#### United States Patent & Trademark Office

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<sup>1</sup> )Patent Application Information PAIR)	12/023,431	FLOW-THROUGH OXYGEN	ATOR	<b>8</b> 9
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Employee & Office Directories Resources & Public Nobces	Application Type:	Utility	Status Date:	02-10-2010
<ul> <li>Resources &amp; Public Notices</li> </ul>	Examiner Name:	ALLEN, CAMERON J	Location: 👁	ELECTRONIC
Patent Searches	Group Art Unit:	1797	Location Date:	-
Patent Official Gazette 11 Search Patents & Applications 11 Search Biological Sequences 11 Copies, Products & Services	Confirmation Number:	7381	Earliest Publication No:	US 2008-0179259 A1
	Attorney Docket Number:	4056.020503	Earliest Publication Date:	07-31-2008
Diher	Class / Subclass:	210/748	Patent Number:	7,670,495
opyrights rademarks slidy 6 Law soorts	First Named Inventor:	James Andrew Senkiw , Minneapolis, MN (US)	Issue Date of Patent:	03-02-2010
	Title of Invention:	DOW-THDOIC	+ OXYGENATOR	

Call the Patent Electronic Business Center at (866) 217-9197 (toll free) or e-mail EBC@uspto.gov for specific questions about Patent Application Information Retrieval (PAIR).
 Send general questions about USPTO programs to the USPTO Contact Center (UCC).
 If you experience technical difficulties or problems with this application, please report them via e-mail to Electronic Business Support or call 1 800-786-9199.

You can suggest USPTO webpages or material you would like featured on this section by E-mail to the webmaster@uspto.gov. While we cannot promise to accommodate all requests, your suggestions will be considered and may lead to other improvements on the website.

Home | Site Index | Search | eBusiness | Help | Privacy Policy

THIS PATENT ISSUED 3/2/2010. In DISCUSSIONS WITH PAUL HAMN, IT GIVES US BREAD COVERAGE.

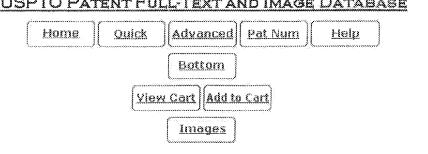
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#### **JA336**

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 339 of 1320

United States Patent: 7670495

Page 1 of 12



USPTO PATENT FULL-TEXT AND IMAGE DATABASE

**United States Patent** Senkiw

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(1 of 1)

Flow-through oxygenator

#### Abstract

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flowthrough model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

Inventors: Senkiw; James Andrew (Minncapolis, MN) Assignce: Oxygenator Water Technologies, Inc. (Minnetonka, MN) Appl. No.: 12/023,431 Filed: January 31, 2008

#### **Related U.S. Patent Documents**

<b>Application</b> Number	<b>Filing Date</b>	Patent Number	<b>Issue Date</b>
10732326	Dec., 2003	7396441	
10372017	Feb., 2004	6689262	
60358534	Feb., 2002		

Current U.S. Class:	204/232; 204/245; 205/628; 210/243; 210/600
<b>Current International Class:</b>	C02F 1/48 (20060101); C02F 1/00 (20060101); C25B
Field of Search:	1/02 (20060101); C25B 1/04 (20060101) 210/748,600,243 204/278,242,243,275.1,232,286.1,554,660
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**JA337** 

Page 2 of 12

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Primary Examiner: Griffin; Walter D Assistant Examiner: Allen; Cameron J Attorney, Agent or Firm: Patterson, Thuente, Skaar & Christensen, P.A.

#### Parent Case Text

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JA338

Page 3 of 12

RELATED APPLICATIONS

This application is a division of application Ser. No. 10/732,326 filed Dec. 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

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Claims	
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The invention claimed is:

1. A method for treating waste water comprising; providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other, placing the emitter within a conduit; and passing waste water through the conduit.

2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breading the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.

5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.

7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.

8. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising: inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.

9. A method for lowering the biologic oxygen demand of polluted water comprising: passing the polluted water through a vessel containing the emitter of claim 2.

10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

#### Page 4 of 12

11. The emitter of claim 2, further comprising a timer control.

12. The emitter of claim 2, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.

Description

## FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

#### BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

TABLE-US-00001 AT THE CATHODE: 4H.sub.2O + 4e.sup.- .fwdarw. 4OH.sup.- + 2H.sub.2 AT THE ANODE: 2H.sub.2O .fwdarw. O.sub.2 + 4H.sup.+ + 4e.sup.- NET REACTION: 6H.sub.2O .fwdarw. 4OH.sup.- + 4H.sup.+ ++ 2H.sub.2 + O.sub.2

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Page 5 of 12

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

# SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

**JA341** 

Page 6 of 12

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

# BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the O.sub.2 emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the O.sub.2 emitter.

FIG. 4 shows a funnel or pyramid variation of the O.sub.2 emitter.

FIG. 5 shows a multilayer sandwich O.sub.2 emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

# DETAILED DESCRIPTION OF THE INVENTION

Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O.sub.2 emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O.sub.2. In the special dimensions of the invention, as explained in more detail in the following examples, O.sub.2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H.sub.2 formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

# EXAMPLE 1

#### Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 8 of 12

shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon.RTM., polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an O.sub.2 emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### **EXAMPLE 2**

#### Measurement of O.sub.2 Bubbles

Attempts were made to measure the diameter of the O.sub.2 bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed O.sub.2 bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

## EXAMPLE 3

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Page 9 of 12

Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H.sub.2) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

 TABLE-US-00002 TABLE I Emitter Model Gallons Volts Amps Max. Ave Watts Bait keeper 5 6 0.090
 0.060 0.36 Livewell 32 12 0.180 0.120 1.44 OEM 2 inch 10 12 0.210 0.120 1.44 Bait store 70 12 0.180

 0.180 2.16 Double cycle 2 12 0.180 0.180 2.16 OEM 3 inch 50 12 0.500 0.265 3.48 OEM 4 inch 80 12
 0.980 0.410 4.92 Water pail 2 24 1.200 1.200 28.80 Plate 250 12 5.000 2.500 30.00

**EXAMPLE 4** 

Multilayer Sandwich O.sub.2 Emitter

An O.sub.2 emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

TABLE-US-00003 cathode screen 0.045 inches thick nylon spacer 0.053 inches thick anode grid 0.035 inches thick nylon spacer 0.053 inches thick cathode screen 0.045 inches thick,

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

**EXAMPLE 5** 

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Page 10 of 12

Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in one-inch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 11 of 12

half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

TABLE-US-00004 TABLE II Control, grams Test, grams tomatoes from tomatoes from eight plants/ seven plants/ Week of: cumulative total cumulative total July 27 240 400 August 3 180 420 2910 3310 August 10 905 1325 1830 5140 August 17 410 1735 2590 7730 August 24 3300 5035 2470 10200 August 31 4150 9175 1580 11780 September 15 not weighed 3710 15490 Final Harvest 6435 15620 8895 24385 September 24

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

#### **EXAMPLE 6**

Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120.degree. angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2.degree. C. but the flowing water cooled slightly to 11 or 11.5.degree. C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE-US-00005 TABLE III ACTIVE DO OF\* ELECTRODE CURRENT, FLOW RATE SAMPLE AT MODEL AREA, SQ.IN. VOLTAGE AMPS. GAL/MINUTE ONE MINUTE 2-Inch "T" 2 28.3 0.72 12 N/A 3-inch "T" 3 28.3 1.75 12 N/A 2-plate Tube 20 28.3 9.1 12 8.4 3-Plate tube 30 28.3 12.8 12 9.6

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Page 12 of 12

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The oneminute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

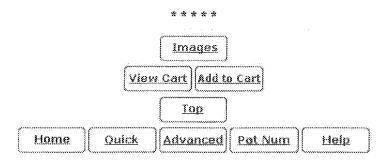
It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

EXAMPLE 7

Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.



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#### AGREEMENT OF STRICT FORECLOSURE

THIS AGREEMENT OF STRICT FORECLOSURE (the "<u>Agreement</u>") is made, entered into and effective as of October 4, 2012 (the "<u>Effective Date</u>"), by and between AQUA INNOVATIONS INCORPORATED ("<u>Debtor</u>"), a Minnesota corporation, and ROY H. LECY ("<u>Secured Party</u>"), who holds a certain security interest in the assets of Debtor as set forth below as security under a certain Promissory Note dated December 1, 2006, executed by Debtor in favor of Secured Party in the principal amount of Two Hundred Eighteen Thousand Eight Hundred Twenty-Seven and 29/100 Dollars (\$218,827.49) (the "<u>Note</u>"). Either Debtor or Secured Party may be individually referred to herein as a "<u>party</u>" or collectively as the "<u>parties</u>."

# RECITALS

WHEREAS, Secured Party is a shareholder of Debtor who holds nine hundred twentyeight thousand three hundred thirty-four (928,334) shares of Debtor's common stock and twentysix thousand six hundred sixty-seven (26,667) shares of Debtor's Preferred Series A stock; and

WHEREAS, Secured Party loaned Debtor various amounts of money over the course of several years, as documented in the Note; and

WHEREAS, Debtor is in default of its obligations pursuant to the Note; and

WHEREAS, as of December 31, 2011, the sum of Two Hundred Seven Thousand Ninety-Eight and no/100 Dollars (\$207,098.00) remains due and owing Secured Party from Debtor (the "Outstanding Debt"); and

WHEREAS, the Note provides Secured Party a security interest in all of Debtor's "patents and physical assets"; and

WHEREAS, Secured Party perfected his security interest via the filing of a Uniform Commercial Code Financing Statement with the Minnesota Secretary of State on or about February 10, 2012, Filing Number 201227190568; and

WHEREAS, Secured Party is currently the only secured creditor of Debtor; and

WHEREAS, Debtor is no longer a going business concern; and

WHEREAS, Debtor has no viable assets other than its rights as Licensee under that certain License Agreement dated July 30, 2008 (the "License Agreement"), a copy of which is attached hereto as Exhibit A, entered into by and between Debtor and Oxygenator Water Technologies, Inc. ("OWT"), a Minnesota corporation, pursuant to which Debtor holds certain perpetual, exclusive and royalty free licenses as further described in the License Agreement, any property of Debtor as set forth in the License Agreement, including but not limited to any Licensee Documents, Licensee Improvements, Licensee Patents, Licensee Products, Licensee Property or Licensee Technology, all as defined in the License Agreement and certain shares of

Agreement of Strict Foreclosure

**JA349** 

OWT's common stock held by Debtor (the "<u>OWT Stock</u>" or collectively with the License Agreement and the other property of Debtor set forth herein, the "<u>Collateral</u>"); and

WHEREAS, Secured Party, as Debtor's sole secured creditor, is entitled to foreclose on the Collateral securing the Note; and

WHEREAS, Secured Party has agreed and Debtor has consented to Secured Party's acceptance of the Collateral in full satisfaction of Debtor's obligations under the Note in accordance with Sections 9-620 through 9-622 of the Uniform Commercial Code (the "<u>UCC</u>"), as adopted in the State of Minnesota as Minnesota Statutes Sections 336.9-620 through 336.9-622.

#### AGREEMENTS

**NOW, THEREFORE,** in consideration of the foregoing, Debtor and Secured Party hereto agree as follows:

1. <u>Recitals</u>. Debtor hereby acknowledges that the recitals set forth above are true and correct and such recitals are hereby made a part of this Agreement.

2. <u>Conveyance of Collateral; Satisfaction of Outstanding Debt</u>. Pursuant to Minnesota Statutes Sections 336.9-620 through 336.9-622, Debtor assigns all right, title and interest in and to the Collateral to Secured Party, agrees to immediately surrender the Collateral to Secured Party and Secured Party shall retain the Collateral in full satisfaction of the Outstanding Debt. Debtor shall deliver the Stock Powers Certificate (Assignment Separate from Certificate) for the OWT Stock attached hereto as <u>Exhibit B</u> to Secured Party contemporaneous with Debtor's execution of this Agreement. Secured Party does <u>not</u> assume, and nothing herein shall be construed to obligate Secured Party to pay, any leases, liabilities or obligations of Debtor.

**3.** <u>Waiver</u>. Pursuant to Section 9-624 of the UCC, Minnesota Statutes Section 336.9-624, Debtor hereby waives and renounces all of its rights to notification under Section 9-611 of the UCC, Minnesota Statutes Section 336.9-611, or any other state in which any Collateral may be located as to the sale or other disposition by Secured Party of the Collateral and all rights under Sections 9-620 and 9-623 of the UCC, Minnesota Statutes Sections 336.9-620 and 336.9-623, regarding acceptance of collateral as discharge of the obligations of Debtor to Secured Party, mandatory disposition of the Collateral and the waiver of Debtor's rights to redeem collateral, respectively. Debtor further knowingly and intelligently waives any rights it may have to notice and a hearing before a court of competent jurisdiction.

#### 4. <u>Representations and Warranties</u>. Debtor represents and warrants the following:

4.1 Debtor has the power and is duly authorized to enter into and perform this Agreement, and Debtor has complied with and is in good standing with respect to all laws, statutes and ordinances of all federal, state and local governmental entities having jurisdiction over them. Debtor hereby represents and warrants that this Agreement is a legal, valid and binding agreement, enforceable in accordance

Agreement of Strict Foreclosure

**JA350** 

with its terms and shall be binding upon Debtor and its respective representatives, successors and assigns.

4.2 Debtor represents, warrants and covenants that it has valid title to all of the Collateral being turned over herewith.

5. [Intentionally Omitted].

6. <u>Representation by Counsel</u>. Debtor acknowledges that it has been represented by legal counsel of its choice, Jamie R. Pierce of Hinshaw & Culbertson, LLP, in connection with the execution and delivery of this Agreement.

7. <u>Notices</u>. Any notice required or permitted under this Agreement shall be in writing and shall be deemed to be given when and if sent by certified mail, return receipt requested, postage pre-paid, properly addressed as follows, or such other address as may hereafter be designated in writing by either of the parties:

Secured Party:

Debtor:

Roy Lecy c/o Nathan M. Brandenburg Siegel Brill, P.A. 100 Washington Avenue South, Suite 1300 Minneapolis, MN 55401

Aqua Innovations Incorporated 6101 Baker Road, Suite 206 Minnetonka, MN 55345

With a copy to:

Jamie R. Pierce Hinshaw & Culbertson, LLP 333 South Seventh Street, Suite 2000 Minneapolis, MN 55402

8. <u>Amendments, Waivers, Assignment</u>. No amendment, waiver or assignment of the provisions of this Agreement shall be effective unless the same shall be in writing and be signed by the party against whom it is to be enforced, and then such amendment, waiver or assignment shall be effective only in the specific interest and for the specific purpose which given.

9. <u>Necessary Documents</u>. The parties agree that they shall execute any and all documents necessary to carry out the terms and conditions of this Agreement.

10. <u>Governing Law; Venue</u>. This Agreement shall be governed and construed under and in accordance with the laws of the State of Minnesota. Any dispute arising under this Agreement

Agreement of Strict Foreclosure

**JA351** 

and/or between the parties shall be venued in the state and federal courts located in Minneapolis, Minnesota.

11. <u>Entire Agreement</u>. This Agreement contains the entire Agreement by and between the parties hereto with respect to the transactions contemplated herein, which shall supersede any prior oral negotiations and agreements and shall be binding upon the parties hereto and their successors and assigns.

12. <u>Merger</u>. All prior oral and written communications, commitments, alleged commitments, promises, alleged promises, agreements and alleged agreements by or between Secured Party and Debtor are hereby merged into this Agreement.

13. <u>Severability</u>. If any part of this Agreement is held to be illegal, invalid or unenforceable, the remainder of this Agreement shall continue in full force and effect, notwithstanding such illegality, invalidity or unenforceability.

14. <u>Headings</u>. The section headings in this Agreement are included herein for convenience or reference only and shall not constitute a part of this Agreement for any other purposes.

15. <u>Successors and Assigns</u>. This Agreement shall be binding upon and shall inure to the benefit of the parties hereto and their respective successors and assigns, except that Debtor may not assign or transfer its rights or obligations hereunder without the prior written consent of Secured Party.

16. <u>Counterparts</u>. This Agreement may be executed in one or more counterparts, each of which shall be deemed to be an original and all of which shall constitute one and the same instrument.

17. <u>Indemnification</u>. Debtor agrees to indemnify, defend and hold Secured Party, his respective employees, agents, representatives and attorneys harmless from any claim or cause of action (except for a claim of fraud against Secured Party) by any third party based in whole or in part upon the terms of this Agreement or their actions or omissions in fulfilling or enforcing this Agreement or based upon any failure by Debtor to pay all taxes, or other indebtedness or fulfill any obligations they may have with other third parties which may be affected by this Agreement or the omissions or actions of the parties relative thereto.

[This space intentionally left blank; signature page follows.]

Agreement of Strict Foreclosure

**JA352** 

IN WITNESS WHEREOF, Secured Party and Debtor hereto have executed this Agreement as of the Effective Date.

Debtor:

**Aqua Innovations Incorporated** 

By: Its: EXECUTIVS OHITER

STATE OF MINNESOTA ) ) ss. COUNTY OF HENNEPIN )

This. document was acknowledged before me on October <u>4</u>, 2012, by <u>Anel Jush</u>, as <u>hig Cleantive of Jush</u> of Aqua Innovations Incorporated, a Minnesota corporation.

Notary Stamp:



Notary Signature  $\checkmark$ 

Roy H. Lecy

STATE OF MINNESOTA ) ) ss. COUNTY OF HENNEPIN )

This document was acknowledged before me on September \_\_\_\_\_, 2012, by Roy H. Lecy, an individual resident of the State of Minnesota.

Notary Stamp:

Notary Signature

M:\26141-001\Strict Foreclosure Agreement 05

Agreement of Strict Foreelosure

5

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

IN WITNESS WHEREOF, Secured Party and Debtor hereto have executed this Agreement as of the Effective Date.

**Aqua Innovations Incorporated** Debtor: By: \_\_\_\_\_\_ Its: \_\_\_\_\_ STATE OF MINNESOTA SS. ) COUNTY OF HENNEPIN ) This document was acknowledged before me on September \_\_\_\_, 2012, by \_\_\_\_\_, as \_\_\_\_\_\_ of Aqua Innovations Incorporated, a Minnesota corporation.

Notary Stamp:

Secured Party:

248	eag
Roy H. Lecy	9

)

STATE OF MINNESOTA COUNTY OF HENNEPIN )

SS.

Cotober This document was acknowledged before me on September <u>8</u>, 2012, by Roy H. Lecy, an individual resident of the State of Minnesota.

Notary Signature

Notary Stamp:



M:\26141-001\Strict Foreclosure Agreement 05

Agreement of Strict Foreclosure

Jaui K. Haurt Notary Signature

5

**OWT Ex. 2118** Tennant Company v. OWT IPR2021-00625

Page 356

# <u>EXHIBIT A</u> LICENSE AGREEMENT

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

# EXHIBIT A LICENSE AGREEMENT

# AMENDMENT TO LICENSE AGREEMENT

THIS AMENDMENT TO LICENSE AGREEMENT (the "Amendment"), is made, entered into and effective as of October 4, 2012, by and between OXYGENATOR WATER TECHNOLOGIES, INC. ("Licensor"), a Minnesota corporation doing business as Water D.O.G. Works, and ROY H. LECY ("Lecy"), and amends certain terms of that certain License Agreement dated July 30, 2008 (the "License Agreement"), entered into by and between Licensor and Aqua Innovations Incorporated ("Aqua"), a Minnesota corporation. Licensor and Lecy may be individually referred to herein as a "party" or collectively as the "parties."

WHEREAS, Licensor, as Licensor, and Aqua, as Licensee, entered into the License Agreement on or about July 30, 2008, a copy of which is attached hereto as <u>Exhibit A</u>; and

WHEREAS, pursuant to an Agreement of Strict Foreclosure dated October 4, 2012, Aqua conveyed all right, title and interest it held in the License Agreement to Lecy; and

WHEREAS, the parties desire to formally amend the License Agreement to reflect Lecy's interest in the Agreement via this Amendment.

# NOW THEREFORE, it is hereby agreed as follows:

1. <u>Incorporation of Recitals</u>. The recitals set forth above are true and correct and incorporated as if fully stated herein.

2. <u>Amendment of License Agreement</u>. All references in the License Agreement to Licensee shall mean Roy H. Lecy and not Aqua. Licensor hereby acknowledges the acquisition by Lecy of all right, title and interest of Aqua in the License Agreement and further acknowledges and agrees that the License Agreement is a binding contract in full force and effect and that Lecy may assign his interest therein at will.

3. <u>Remaining Terms in Full Force and Effect</u>. No other terms of the License Agreement or any schedule or exhibit thereto shall be amended or modified in any way and the License Agreement shall remain in full force and effect as amended via this Amendment and the parties hereby reaffirm their respective obligations thereunder.

Oxygenator Water Technologies, Inc.

By: . Its 10-84-2012

Roy H, Lee

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA357

2

# PATENT ASSIGNMENT

# Electronic Version v1.1 Stylesheet Version v1.1

SUBMISSION TYPE:		NEW ASSIC	GNMENT		
NATURE OF CONVEYANCE:		LICENSE			
CONVEYING PARTY DATA					
Name Execution Date					
Roy H Lecy				10/04/2012	
RECEIVING PARTY	DATA				
Name:	O2 Marine Techr	ologies, Inc.			
Street Address:	6651 Hazeltine B	oulevard			Ì
City:	Excelsior				
State/Country:	MINNESOTA				
Postal Code:	55331				]
Property Type Number				]	
			Number		
Patent Number:		9262			
Patent Number:	739	6441			
Patent Number:	767	0495			
CORRESPONDENC	E DATA				
Fax Number:	6123396591	when the fax of	Hampt in unquanantul		
Phone:	6123376100	when the lax at	ttempt is unsuccessful.		
Email: nathanbrandenburg@siegelbrill.com					
Correspondent Name: Nathan M. Brandenburg					
Address Line 1: 100 Washington Avenue South					
Address Line 2: Suite 1300					
Address Line 4: Minneapolis, MINNESOTA 55401					
ATTORNEY DOCKE	T NUMBER:	25991-002			
NAME OF SUBMITTER:		Nathan M. E	Nathan M. Brandenburg		
Signature:		/nathanmbra	andenburg/		

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 361 of 1320

Date:	04/30/2013
	This document serves as an Oath/Declaration (37 CFR 1.63).
Total Attachments: 5 source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page source=Patent Sublicense Agreement#page	92.tif 93.tif 94.tif

#### PATENT SUBLICENSE AGREEMENT

THIS PATENT SUBLICENSE AGREEMENT (this "<u>Agreement</u>") is made, entered into and effective as of October 4, 2012 (the "<u>Effective Date</u>") by and between ROY H. LECY ("<u>Sublicensor</u>"), a Minnesota resident, and O2 MARINE TECHNOLOGIES, INC. ("<u>Sublicensee</u>"), a Minnesota corporation. Sublicensor or Sublicensee may be individually referred to herein as a "<u>party</u>" or collectively as the "<u>parties</u>."

WHEREAS, Sublicensor is the holder of certain patent licenses as set forth in a certain License Agreement dated July 30, 2008, entered into by and between Oxygenator Water Technologies, Inc. ("<u>QWT</u>"), as Licensor, and Aqua Innovations Incorporated ("<u>Aqua</u>"), as Licensee (the "<u>License Agreement</u>"), as amended via a certain Amendment to License Agreement dated October 4, 2012 (the "<u>Amendment</u>" or collectively with the License Agreement, the "<u>License Agreement</u>"), pursuant to which Roy H. Lecy acquired all right title and interest of Aqua in the License Agreement; and

WHEREAS, copies of the License Agreement and Amendment are attached hereto as Exhibit A.

NOW THEREFORE, the parties hereby agree as follows:

## ARTICLE ONE GRANT OF SUBLICENSE

1.1 License. Sublicensor warrants he holds a valid license pursuant to the License Agreement for United States Patent Number 6,689,262 B2 issued on February 10, 2004, entitled "Microbubbles of oxygen," United States Patent Number 7,396,441 B2 issued on July 8, 2008, entitled "Flow-through oxygenator," and United States Patent Number 7,670,495 issued on March 2, 2010, entitled "Flow-through oxygenator" (collectively, the "Licensed Patents" or each a "Licensed Patent").

1.2 <u>Grant of Sublicense</u>. Effective upon execution of this Agreement, and for consideration stated in Article Three of this Agreement, Sublicensor grants to Sublicensee a royalty based, limited, exclusive sublicense to make, sell, and offer for sale products covered by the Licensed Patents in the United States and to sell and offer for sale in any country products covered by the Licensed Patents (the "Licensed Products"), subject to the limitations set forth in this Agreement. The Licensed Patents are sublicensed "As Is" and without warranty of any kind by Sublicensor.

## ARTICLE TWO LICENSED FIELD

2.1 <u>Licensed Field</u> Sublicensee may sell and offer for sale only Licensed Products manufactured for retail sale and use in the sport fishing industry (the "<u>Licensed Field</u>") and for no other purposes. Sublicensee hereby agrees to offer for sale and to sell only the Licensed Products in the Licensed Field. Sublicensee acknowledges and agrees that if Sublicensee offers for sale or sells the Licensed Products outside the Licensed Field such activity by Sublicensee will be deemed a material breach of this Agreement and Sublicensor shall have the right to terminate this Agreement without notice.

1

## ARTICLE THREE ROYALTY

**3.1** <u>Royalty</u>. Sublicensee shall pay royalties to Sublicensor at a rate of five percent (5%) of the net receipts realized by Sublicensee upon any Licensed Products sold by Sublicensee. For purposes of this Agreement, "net receipts" shall mean gross sale proceeds, less cost of goods sold, freight, discounts offered by Sublicensee, returns and other costs or expenses incurred by Sublicensee in the manufacture and sale of the Licensed Products. Any royalties shall be paid to Sublicensor on a calendar quarterly basis, sixty (60) days after the end of each calendar quarter (May 30, August 30, December 30 and March 1 or 2).

Accounting. For all Licensed Products sold by Sublicensee, Sublicensee will account to 3.2 Sublicensor on a calendar quarterly basis, indicating the number of Licensed Products sold within thirty (30) days following the end of each calendar quarter for the term hereof. Sublicensee shall make such accountings to Sublicensor via paper or electronic accounting statements in a mutually accepted compatible format. Sublicensee agrees to keep and maintain true and accurate records and books of account in connection with all sales related to any such products and all transactions related thereto or otherwise contemplated under this Agreement, and shall retain all such records and books for a period of not less than five (5) years after each accounting to Sublicensor. Sublicensor, by its designated representative, shall have the right, upon reasonable written notice, and during normal office hours, to examine the books and records of Sublicensee, as the same pertain to the subject matter of this Agreement, and to make copies and extracts thereof. Sublicensee shall cooperate with Sublicensor's representatives to assist them in understanding all such material. If, as a result of any audit, it is determined that Sublicensee has understated the royalties due to Sublicensor by ten percent (10%) or more, Sublicensee shall pay to Sublicensor the amount by which royalties have been understated plus a ten percent (10%) penalty fee, and shall reimburse Sublicensor for the cost of the audit.

## ARTICLE FOUR PACKAGING

4.1 <u>Packaging</u>. Sublicensec shall use its own tradename or trade or servicemark on the packaging for the Licensed Products. In no event shall Sublicensee use any of Sublicensor's trademarks on the packaging for the Licensed Products.

## ARTICLE FIVE LICENSED PRODUCT REVIEW

5.1 Licensed Product Review. Sublicensor shall have the right to review the Licensed Products and packaging for the Licensed Products to be sold or offered for sale by Sublicensee prior to first sale of the Licensed Products and thereafter on a quarterly basis. Sublicensor shall have the right to provide suggestions concerning the quality and design of the Licensed Products, including packaging, to be sold or offered for sale by Sublicensee. If Sublicensor determines the quality of Licensed Products and/or packaging on the Licensed Products is unacceptable to Sublicensor, Sublicensor shall work with Sublicensee and offer suggestions to make the Licensed Products suitable and ready for market. Sublicensor and Sublicensee shall be willing to mutually work together in a reasonable manner without undue restriction.

2

**JA361** 

## <u>ARTICLE SIX</u> MARKETING

6.1 <u>Marketing</u>. Sublicensee shall mark all Licensed Products made, used or sold under the terms of this Agreement with the following: "Protected by U.S. Patent Nos. 6,689,262, 7,396,441 and 7,670,495. Other Patents Pending."

## ARTICLE SEVEN TERM

7.1 <u>Term</u>. The sublicense so granted pursuant to this Agreement shall be effective from the Effective Date and shall terminate with respect to a Licensed Patent on the expiration of the Licensed Patent, subject to Section 7.2 of this Agreement.

7.2 <u>Termination</u>. Notwithstanding Section 7.1 of this Agreement, this Agreement shall terminate:

- 7.2.1 Upon the mutual agreement of the parties;
- 7.2.2 Upon ten (10) days' written notice from Sublicensor to Sublicensee upon Sublicensee's failure to pay any royalty due and owing Sublicensor;
- 7.2.3 Immediately if Sublicensee files of a petition of bankruptcy, or a petition or answer seeking reorganization, readjustment or rearrangement of its business or affairs under any law or governmental regulation relating to bankruptcy or insolvency;
- 7.2.4 Immediately if in Sublicensor's reasonable business judgment Sublicensee undertakes any action that derogates, disparages or impairs any of the Licensed Patents;
- 7.2.5 Immediately upon the terms of Section 2.1 of this Agreement; or
- 7.2.6 Upon ninety (90) days' written notice from Sublicensor to Sublicensee if Sublicensee fails to actively sell any Licensed Products or otherwise fails to actively and diligently utilize the sublicense granted by this Agreement.

## <u>ARTICLE EIGHT</u> INFRINGEMENT

8.1 <u>Notice</u>. Sublicensee shall inform Sublicensor within thirty (30) days and in writing of any alleged infringement of the Licensed Patents by a third party.

8.2 <u>Legal Action: Mutual Cooperation</u>. Sublicensor shall have the right, but shall not be obligated, to prosecute at its own expense any infringement of the Licensed Patents and, in furtherance of such right, Sublicensee hereby agrees that Sublicensor may, if required by law or otherwise, include Sublicensee as a party plaintiff in any such suit, without expense to

3

JA362

Sublicensee. The total cost of any such infringement action commenced or defended solely by Sublicensor shall be borne by Sublicensor, and Sublicensor shall keep any recovery or damages. In the event that any action alleging invalidity, non-enforceability, or non-infringement of any of the Licensed Patents shall be brought by a third party, Sublicensor, at its option, shall have the right to defend such actions.

## ARTICLE NINE INDEMNIFICATION

9.1 <u>Indemnification</u>. Sublicensee shall indemnify, defend, and hold harmless Sublicensor from any and all actions, claims, suits, losses, liabilities, damages, costs, fees, and expenses (including attorney fees) resulting from or arising out of the exercise of the Sublicensee's rights granted under this Agreement. This indemnification shall include, but is not limited to, any and all actions alleging product liability, patent infringement, or other type of intellectual property matter.

## ARTICLE TEN GENERAL TERMS

10.1 <u>Assignment</u>. The rights and sublicenses granted by Sublicensor in this Agreement are personal to Sublicensee and may not be assigned or otherwise transferred without the written consent of Sublicensor. Sublicensor may provide such consent upon request from Sublicensee for any assignment to a third party who is acquiring substantially all of the business assets of Sublicensee, but Sublicensor reserves the right to deny consent if the third party is a competitor of Sublicensor.

10.2 <u>Sublicense</u>. The rights and license granted by Sublicensor in this Agreement may not be sublicensed by Sublicensee without the written consent of Sublicensor.

10.3 <u>Confidentiality</u>. Sublicensor and Sublicensee both agree the terms of this Agreement are confidential and shall not be disclosed to any third party.

10.4 <u>Governing Law</u>. This Agreement shall be construed and enforced according to the laws of the State of Minnesota. Any disputes arising out of, under, or relating to the negotiation, drafting, execution, validity, interpretation, breach, or enforcement of this Agreement shall be venued in the state or federal courts located in Minneapolis, Minnesota.

10.5 <u>Entire Agreement</u>. Sublicensee and Sublicensor acknowledge receipt of this Agreement and agree that with respect to the subject matter hereof this Agreement is the entire agreement of the parties and supersedes any previous oral or written communications or understandings, and that each provision has been given due consideration and accepted without duress.

10.6 <u>Attorneys' Fees</u>. In the event that any proceeding, suit or action is brought by any party under this Agreement to enforce any of its terms, it is agreed that the prevailing party shall be entitled to reasonable attorneys' fees to be fixed by the trial and appellate courts in any such proceeding or as incurred in the collection of any judgment.

4

JA363

10.7 Counterparts. This Agreement may be signed in counterparts by the parties hereto with the same force and effect as if the above parties signed the same original agreement. Facsimile and electronic copies and photocopies of the parties' signatures to this Agreement shall be valid and enforceable to the same extent as original signatures and the parties hereby waive any requirement that original signatures be produced as a condition of proving the validity of or otherwise enforcing this Agreement

IN WITNESS WHEREOF, Sublicensor and Sublicensee have executed this Agreement as of the Effective Date.

Sublicensor:

Sublicensee:

Roy H. Lecy

02 MARINE TECHNOLOGIES, INC.

By Dennis Clark

Its: President

5

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

**JA364** 

# PATENT ASSIGNMENT

# Electronic Version v1.1 Stylesheet Version v1.1

SUBMISSION TYPE	Ξ:		NEW ASSIGNMENT		
NATURE OF CONV	'EYANCE:		ASSIGNMENT		
CONVEYING PART	Ύ DATA				
		N	Name	Execution Date	
Aqua Innovations, I	nc.			10/04/2012	
RECEIVING PARTY	/ DATA				
Name:	Roy H Lecy				
Street Address:	2640 North S	Saunde	ers Lake Drive		
City:	Minnetrista				
State/Country:	MINNESOTA	4			
Postal Code:	55364				
PROPERTY NUMB	ERS Total: 3				
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Property	Туре	Number			
Patent Number:	6689262				
Patent Number:		73964	396441		
Patent Number:		76704	495		
CORRESPONDENC	CE DATA				
Fax Number:	612339				
	<i>II be sent via US</i> 612-337		hen the fax attempt is unsuccessful.		
Phone: Email:					
Correspondent Nar	nathanbrandenburg@siegelbrill.com ame: Nathan M. Brandenburg				
Address Line 1:	· •				
Address Line 2:	0				
Address Line 4:					
ATTORNEY DOCKET NUMBER:		26141-001			
NAME OF SUBMITTER:		Nathan M. Brandenburg			
Signature:		/nathanmbrandenburg/			

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 368 of 1320

Date:	04/30/2013
	This document serves as an Oath/Declaration (37 CFR 1.63).
Total Attachments: 27	
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# AMENDMENT TO LICENSE AGREEMENT

THIS AMENDMENT TO LICENSE AGREEMENT (the "<u>Amendment</u>"), is made, entered into and effective as of October 4, 2012, by and between OXYGENATOR WATER TECHNOLOGIES, INC. ("<u>Licensor</u>"), a Minnesota corporation doing business as Water D.O.G. Works, and ROY H. LECY ("<u>Lecy</u>"), and amends certain terms of that certain License Agreement dated July 30, 2008 (the "<u>License Agreement</u>"), entered into by and between Licensor and Aqua Innovations Incorporated ("<u>Aqua</u>"), a Minnesota corporation. Licensor and Lecy may be individually referred to herein as a "party" or collectively as the "<u>parties</u>."

WHEREAS, Licensor, as Licensor, and Aqua, as Licensee, entered into the License Agreement on or about July 30, 2008, a copy of which is attached hereto as <u>Exhibit A</u>; and

WHEREAS, pursuant to an Agreement of Strict Foreclosure dated October 4, 2012, Aqua conveyed all right, title and interest it held in the License Agreement to Lecy; and

WHEREAS, the parties desire to formally amend the License Agreement to reflect Lecy's interest in the Agreement via this Amendment.

# NOW THEREFORE, it is hereby agreed as follows:

1. <u>Incorporation of Recitals</u>. The recitals set forth above are true and correct and incorporated as if fully stated herein.

2. <u>Amendment of License Agreement</u>. All references in the License Agreement to Licensee shall mean Roy H. Lecy and not Aqua. Licensor hereby acknowledges the acquisition by Lecy of all right, title and interest of Aqua in the License Agreement and further acknowledges and agrees that the License Agreement is a binding contract in full force and effect and that Lecy may assign his interest therein at will.

3. <u>Remaining Terms in Full Force and Effect</u>. No other terms of the License Agreement or any schedule or exhibit thereto shall be amended or modified in any way and the License Agreement shall remain in full force and effect as amended via this Amendment and the parties hereby reaffirm their respective obligations thereunder.

Oxygenator Water Technologies, Inc.

By: . Its 10-84-2012

Roy H, Lee

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA367

2

## License Agreement

THIS AGREEMENT ("<u>Agreement</u>") is entered into this 30th day of July, 2008 (the "<u>Effective Date</u>"), by and between Oxygenator Water Technologies, Inc., a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensor</u>") and Aqua Innovations, Inc. a Minnesota corporation with offices at 6101 Baker Rd., #206, Minnetonka, Minnesota, 55435 ("<u>Licensee</u>", and Licensor and Licensee each a "<u>Party</u>" and together the "<u>Parties</u>"). Initially capitalized terms defined in this Agreement shall have the meaning ascribed to them respectively herein.

## WITNESSETH:

LICENSOR owns the technology for which patents have been issued and are pending with respect to electrolytic hydrolysis of water to increase its dissolved oxygen content. A more complete description of said technology, together with a description of the patents issued and currently pending for said technology, is set forth in Article 1 below and in Exhibit "A" attached hereto.

LICENSOR anticipates and intends that it will make additional discoveries and improvements to said technology, some of which may be patentable.

It is further anticipated by the parties that LICENSOR may make improvements to said technology and additional discoveries concerning other applications for said technology.

The parties desire that LICENSOR grant a perpetual, exclusive license to LICENSEE to develop and sell throughout the world certain products utilizing the technology LICENSOR has developed and may in the future develop, all according to the terms and conditions set forth in this Agreement.

The parties further desire that LICENSOR will retain the complete and entire right to develop and sell throughout the world in markets not licensed to LICENSEE hereunder products utilizing the technology LICENSOR has developed and may in the future develop or the technology that LICENSEE may develop in the future, also according to the terms and conditions set forth in this Agreement.

Thus, the parties have agreed to enter into a licensing arrangement by which each party will be entitled to benefit from the other party's patents, technology and know-how concerning electrolytic hydrolysis of water in the sale of products in certain markets.

**NOW, THEREFORE,** based on the foregoing and the mutual covenants and agreements herein contained, the parties hereby covenant and agree as follows:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## EXHIBIT "B"

## **LICENSEE Markets**

All worldwide markets for:

- Waste Water Treatment
- Medical Applications
- Sport Fishing
- Aqua Culture
- Horticulture (consumer and commercial)
- Hydroponics

Markets excluded from license agreement (including but not limited to):

- Water Treatment (all applications except waste water)
- Fermentation
- Desalination
- Human Nutrition
- Animal Nutrition

## ARTICLE 1 DEFINITIONS

When used in this Agreement, the following terms have the meanings set forth below unless a different and common meaning of the term is clearly indicated by the context, and variants and derivatives of the following terms shall have correlative meanings:

"Agreement" has the meaning set forth in the preamble.

"LICENSOR Documents" has the meaning set forth in Section 2.6.

"LICENSOR Improvements" means all developments LICENSOR may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable, and which are invented, developed, discovered or otherwise acquired by LICENSOR and which LICENSOR may lawfully communicate to LICENSEE.

"*LICENSOR Markets*" means all uses for the LICENSOR Technology and the LICENSEE Technology other than in the LICENSEE Markets.

"LICENSOR Patents" means all of LICENSOR's patents (whether issued to LICENSOR or controlled by license rights or otherwise and whether such rights are held alone or jointly with others, and patents pending now, or during the term of this Agreement, issued to LICENSOR (by any country) relating to the LICENSOR Technology, including, but not limited to, those patents and those patents pending described on Exhibit A and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSOR Products*" means any product manufactured and/or sold or distributed by LICENSOR or a sub licensee of LICENSOR under any claim contained in the LICENSEE Patents.

"LICENSOR Property" means LICENSOR Patents, LICENSOR Improvements and LICENSOR Technology.

"LICENSOR Technology" means LICENSOR's unpatented technology and information now existing and relating to, and embodying LICENSOR's experience in electrolytic hydrolysis of water. LICENSOR Technology shall include the technical information in all current and future manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSOR to LICENSEE during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSOR imparting the same directly or giving LICENSEE access to any of LICENSOR's production facilities.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA370

"Effective Date" has the meaning set forth in the preamble.

"LICENSEE Documents" has the meaning set forth in Section 2.7.

"LICENSEE Improvements" means all developments LICENSEE may make in the LICENSOR Technology or the LICENSEE Technology prior to the termination of this Agreement, whether or not patentable and which are invented, developed, discovered or otherwise acquired by LICENSEE and which LICENSEE may lawfully communicate to LICENSOR.

"*LICENSEE Markets*" means those markets for Licensee Products as are described in Exhibit B attached hereto.

"LICENSEE Patents" means all of LICENSEE's patents (whether issued to LICENSEE or controlled by license rights or otherwise and whether such rights are held alone or jointly with others) which may after the effective date of this Agreement be issued (by any country) relating to electrolytic hydrolysis of water and any continuations, continuations-in-part, divisions, registrations, confirmations, reissues, renewals or extensions of term thereof.

"*LICENSEE Products*" means any product manufactured and/or sold or distributed to any party other than LICENSOR by LICENSEE or a sublicense of LICENSEE in conformity with the terms of this Agreement, including, but not limited to, any product which is based on any claim or thing contained in any LICENSOR Property.

"LICENSEE Property" means LICENSEE Patents, LICENSEE Improvements and LICENSEE Technology.

"LICENSEE Technology" means LICENSEE's unpatented technology and information which LICENSEE may develop relating to, and embodying LICENSEE's experience in, the manufacturing, the processing, quality control, and sale of the LICENSEE Products. LICENSEE Technology shall include the technical information in all manuals, formulae, specifications, test data and procedures, flow charts, apparatus plans, drawings, designs and other information actually communicated by LICENSEE to LICENSOR during the term of this Agreement, whether contained in documentary form, electronic medium or communicated as a result of LICENSEE imparting the same directly or giving LICENSOR access to any of LICENSEE's production facilities.

"Territory" means the world,

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## ARTICLE 2 MARKETS AND LICENSING

2.1. Exclusive Markets. The parties agree that unless properly terminated by LICENSOR pursuant to Section 5.1 below, LICENSEE will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSEE Markets in the Territory. The parties further agree that LICENSOR will have the exclusive right to exploit the LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSEE Products in the LICENSOR Markets in the Territory. LICENSOR Property and the LICENSEE Property in the manufacture, use and sale or other distribution of LICENSOR Products in the LICENSOR Markets in the Territory. LICENSEE may not, directly or indirectly, distribute in any manner any product which competes with the LICENSEE Products in any manner nor may LICENSEE assist or have any interest in any third party distributing any such products through licensing or assignment of technology to any such third party or by any other means.

2.2. Exclusive License to LICENSEE. Subject to the terms and conditions of this Agreement, LICENSOR hereby confers upon LICENSEE the sole and exclusive license, with the right of sublicense, under the LICENSOR Property, to make, have made, use and sell the LICENSEE Products in the LICENSEE Markets in the Territory and to prevent infringement of the LICENSOR Patents, and to prevent unauthorized use and disclosure of the LICENSOR Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSEE Products.

2.3. Exclusive License to LICENSOR. Subject to the terms and conditions of this Agreement, LICENSEE hereby confers upon LICENSOR the sole and exclusive, royalty-free license, with the right of sublicense, under the LICENSEE Property, to make, have made, use and sell the LICENSOR Products in the LICENSOR Markets in the Territory and to prevent infringement of the LICENSEE Patents, and to prevent unauthorized use and disclosure of the LICENSEE Technology in connection therewith. No license is conferred hereby to make, have made, use and sell articles which are not LICENSOR Products.

**2.4. Product Markings.** The Parties shall insure that all LICENSOR Products and all LICENSEE Products are marked with any applicable patent number and all labeling and other product information shall be marked in such manner as to conform with the patent laws and practices of the country of sale.

2.5 Transfer of Technology by LICENSOR. As promptly as practicable after the execution of this Agreement, LICENSOR shall deliver to LICENSEE all information concerning the LICENSOR Property. LICENSOR also promptly shall deliver to LICENSEE all future information it acquires concerning the LICENSOR Property. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSOR, shall be referred to as "LICENSOR Documents". LICENSEE shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSOR Documents in such manner as to maximize the value of the LICENSOR Patents, the LICENSOR Technology and the LICENSOR Improvements; without limiting the generality of the foregoing, LICENSEE shall, and shall cause its employees and

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSOR Documents. Upon 10 days advance notice and at reasonable times, LICENSOR shall permit LICENSEE access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSOR to answer fully such questions as LICENSEE may have with a view to transferring the LICENSOR Property. Nothing in this Section 2.6 shall require LICENSOR to disclose to LICENSEE any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

## 2.6. Transfer of Technology by LICENSEE. As promptly as practicable after

LICENSEE develops, discovers or otherwise comes into possession of LICENSEE Patents, LICENSEE Improvements and/or LICENSEE Technology, LICENSEE shall deliver to LICENSOR all information concerning same. All documentary information so delivered or any documentary information following non-documentary disclosure by LICENSEE, shall be referred to as "LICENSEE Documents." LICENSOR shall receive, use, maintain, restrict access to or copying of, and safeguard the LICENSEE Documents in such manner as to maximize the value of the LICENSEE Patents, the LICENSEE Technology and the LICENSEE Improvements; without limiting the generality of the foregoing, LICENSOR shall, and shall cause its employees and representatives to, use reasonable care to prevent unauthorized access to, copying, use, publication, disclosure or other dissemination of the LICENSEE Documents. Upon 10 days advance notice and at reasonable times, LICENSEE shall permit LICENSOR access to its technical personnel at its offices or at such locations as is mutually agreed upon by the Parties. During such visits, technically competent personnel will be provided by LICENSEE to answer fully such questions as LICENSOR may have with a view to transferring to LICENSOR the LICENSEE Property. Nothing in this Section 2.7 shall require LICENSEE to disclose to LICENSOR any technological information which it does not own or that is otherwise subject to restrictions on use or disclosure.

2.7. Further Prosecution of Patents. LICENSOR will continue with the prompt prosecution of all pending patent applications filed by LICENSOR as detailed on Schedule "A", so long as it is commercially reasonable to do so, and LICENSOR will periodically advise LICENSEE of the status of such prosecutions. As soon as practical, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSOR Patents. In the event that LICENSEE files an application for a patent(s) covering electrolytic hydrolysis of water, LICENSEE will periodically advise LICENSOR of the status of the prosecution of any such patent. As soon as practical after any such application by LICENSEE, the Parties will confer to determine the countries for which the Parties desire protection for the LICENSEE Patents. From the date of this Agreement, all expenses incurred in filing for and maintaining protection in those countries mutually agreed upon (other than expenses of prosecuting the original patent application in the first jurisdiction, which will be the responsibility of the Party filing the patent application) will be shared equally by the Parties. Either Party may seek protection in any country not mutually agreed upon by paying the full amount of the cost thereof. A party seeking such additional protection will receive the full cooperation of the other Party (other than in paying the expenses thereof) in protecting all patents in any such other country.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA373

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2.8. Additional Covenants. Each of LICENSOR and LICENSEE shall faithfully comply with their respective obligations under this Agreement and shall incorporate all terms and conditions required by this Agreement in any contracts with third parties to whom access to the LICENSOR Property or the LICENSEE Property, as the case may be, may (but only in accordance with this Agreement) be given. Each of LICENSOR and LICENSEE shall indemnify and hold harmless the other Party and its successors and assigns from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on such Party, and arising out of or in connection with or resulting from the marketing, sale or use of the imdemnifying Party's product(s), including any advertising or other promotional activities related thereto.

**2.9. Infringement Actions.** Neither LICENSOR nor LICENSEE will have any responsibility to the other Party for any damage or expense incurred by such other Party which arises from any action, claim or cause of action brought by any person as the result of any alleged patent infringement or trade secret misappropriation by reason of such other Party's manufacture, use or sale of any product under any of the licenses conferred hereby.

2.10. LICENSEE's Rights in Event of Third Party Infringement. LICENSEE shall have the right, in LICENSOR's name (if required by law, otherwise, in LICENSEE's name) but at LICENSEE's sole expense, to sue third parties in the LICENSEE Markets for infringements of the LICENSOR Patents and misappropriation of the LICENSOR Technology and unpatented LICENSOR Improvements, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. LICENSOR may, if it so elects, join in any such suit as a plaintiff. All damages, awards or settlement proceeds in such suit shall be LICENSEE's. If LICENSEE, after notice from LICENSOR of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSOR, in its own name (or, if required by law, in its and LICENSEE's name) and at its own expense, may sue therefore, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSOR shall promptly reimburse LICENSEE for said suitassociated direct expenses upon presentation of LICENSEE's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's.

2.11. LICENSOR's Rights in Event of Third Party Infringement. LICENSOR

shall have the right, in LICENSEE's name (if required by law, otherwise, in LICENSOR's name) but at LICENSOR's sole expense, to sue third parties in the LICENSOR Markets for infringements of the LICENSEE Patents and misappropriation of the LICENSEE Technology and unpatented LICENSEE Improvements, and LICENSEE shall, but at LICENSOR's expense for LICENSEE's direct associated expenses, fully and promptly cooperate and assist LICENSOR in connection with any such suit. LICENSEE may, if it so elects, join in any such suit as a plaintiff. LICENSOR shall promptly reimburse LICENSEE for said suit-associated direct expenses upon presentation of LICENSEE's itemized

statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSOR's. If LICENSOR, after notice from LICENSEE of an alleged infringement or misappropriation, shall within 90 days fail to institute suit, LICENSEE, in its own name (or, if required by law, in its and LICENSOR's name) and at its own expense, may sue therefore, and LICENSOR shall, but at LICENSEE's expense for LICENSOR's direct associated expenses, fully and promptly cooperate and assist LICENSEE in connection with any such suit. LICENSEE shall promptly reimburse LICENSOR for said suit-associated direct expenses upon presentation of LICENSOR's itemized statement therefor. All damages, awards or settlement proceeds in such suit shall be LICENSEE's.

**2.12. LICENSEE Royalty Payment.** None. License is granted without cost to LICENSEE.

## ARTICLE 3 INDEMNIFICATION

**3.1.** Indemnification by LICENSEE. LICENSEE shall indemnify and hold LICENSOR and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSOR arising out of or in connection with or resulting from the marketing, sale or use of the LICENSEE Products, including any advertising or other promotional activities related thereto. LICENSOR shall be an added insured party to LICENSEE's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSEE shall procure and have in place no later than the date on which LICENSEE first makes a delivery of any of the LICENSEE Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSOR is provided at least thirty (30) days advance written notice.

**3.2. Indemnification by LICENSOR.** LICENSOR shall indemnify and hold LICENSEE and its successors and assigns harmless from any injury, loss, or damage of any kind or nature, or any other liability sought to be imposed on LICENSEE arising out of or in connection with or resulting from the marketing, sale or use of the LICENSOR Products, including any advertising or other promotional activities related thereto. At such time, if any, as LICENSOR shall sell LICENSOR Products, LICENSOR shall add LICENSEE as an added insured party to LICENSOR's product liability insurance, which shall have coverage limits of at least two million dollars (\$2,000,000) per incident and which LICENSOR shall procure and have in place no later than the date on which LICENSOR first makes a delivery of any of the LICENSOR Products. Such policy of insurance shall provide that it may not be cancelled unless LICENSEE is provided at least thirty (30) days advance written notice.

## ARTICLE 4 CONFIDENTIALITY

4.1. Restrictions on Use and Disclosure of LICENSOR Property by LICENSEE. LICENSEE shall use the LICENSOR Property in confidence and shall not

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA375

disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate: (a) if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSEE, or (b) upon the expiration of the obligation of LICENSEE under this Agreement to pay royalties to LICENSOR.

4.2. Restrictions on Use and Disclosure of LICENSEE Property by LICENSOR. LICENSOR shall use the LICENSEE Property in confidence and shall not disclose same to its employees to whom access may be given in accordance with this Agreement until each such employee shall have previously agreed not to disclose such information. Restrictions on use and disclosure of any portion thereof shall terminate if that portion is, or becomes, generally known within the related trade or industry through no default of LICENSOR.

## 4.3. Employees; Third Parties Etc. In order to faithfully perform their

respective obligations under sections 4.1 and 4.2, the Parties shall limit access to the other Party's Property to only those of its officers, employees and agents who shall have a need to receive or have access to that portion, and then only for the purposes of the practice under the licenses conferred by this Agreement. Each Party will require any third party, to whom access may be authorized under this Agreement, to execute an appropriate confidentiality agreement.

4.4. Authorized Required Disclosures. Nothing in this Article 4 shall prevent a Party: (a) from complying (but only to the narrowest extent required by law and regulation and with due notice on any submissions to governmental agencies of the confidential or proprietary status of the information with a view toward restricting access to, and use or disclosure by, third parties) with reasonable requirements of governmental agencies to disclose information in order to receive legally required consents or permissions to manufacture or sell that Party's Products; or (b) from disclosing information under court order, but only after having made all reasonable efforts to secure the court's order to (i) limit production, use and disclosure of said information for the purposes of the case and to the narrowest class of disclosures practicable under the circumstances and (ii) hold all proceedings in camera with a sealed record.

## ARTICLE 5 RESOLUTION OF DISPUTES

All claims, disputes and other matters in question arising out of, or relating to, this Agreement or the performance thereof shall be submitted to, and determined by, arbitration if good faith negotiations between the parties do not resolve such claim, dispute or other matter within 60 days. Such arbitration shall proceed in accordance with the Commercial Arbitration Rules of the American Arbitration Association then pertaining (the "Rules"), insofar as such Rules are not inconsistent with the provisions expressly set forth in this Agreement, unless the parties mutually agree otherwise, and pursuant to the following procedures:

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

2 A.

(a) Notice of the demand for arbitration shall be filed in writing with the other Member and with the American Arbitration Association. Each Member shall appoint an arbitrator, and those party-appointed arbitrators shall appoint a third neutral arbitrator within 10 days. If the party-appointed arbitrators fail to appoint a third, neutral arbitrator within 10 days, such third, neutral arbitrator shall be appointed by the American Arbitration Association in accordance with the Rules. A determination by a majority of the panel shall be binding.

(b) Reasonable discovery shall be allowed in arbitration.

(c) All proceedings before the arbitrators shall be held in Minneapolis, Minnesota. The governing law shall be as specified in Section 8.1 below.

(d) The costs and fees of the arbitration, including attorneys' fees, shall be allocated by the arbitrators.

(e) The award rendered by the arbitrators shall be final and judgment may be entered in accordance with applicable law and in any court having jurisdiction thereof.

#### ARTICLE 6 NOTICES

**6.1.** Notices. All communications, demands, notices or objections required or permitted to be given or served under this Agreement shall be in writing and shall be deemed to have been duly given or made only if delivered in person, deposited in the United States mail, postage prepaid, for mailing by certified or registered mail, return receipt requested, or delivered by prepaid overnight courier service, addressed to the appropriate party as follows:

- If to LICENSOR: Richard Disrud, COO Aqua Innovations, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435
- If to LICENSEE: Jeffrey Brink, CEO Oxygenator Water Technology, Inc. 6101 Baker Rd., #206 Minnetonka, Minnesota 55435

Either party may change its address by giving notice in writing, stating the new address, to the other Party as provided in the foregoing manner. Commencing on the tenth (10th) day after the giving of such notice, such newly designated address shall be such Party's address for the purpose of all communications, demands, notices or objections required or permitted to be given or served under this Agreement.

## ARTICLE 7 MISCELLANEOUS

7.1. Governing Law; Court Proceedings. The validity, performance, and all matters relating to the interpretation and effect of this Agreement shall be governed by the internal law in effect in the State of Minnesota without regard to principles of law (such as "conflicts of law") that might make the law of some other jurisdiction applicable. Without limiting the terms set forth in Article 6 with respect to the resolution of disputes, each Party agrees to the exclusive and irrevocable jurisdiction of the federal and state courts of Minnesota for any claim, action or cause of action arising out of or in any way related to this Agreement which may be brought in a court of law and both parties agree that personal service from any such court may be effectively served upon a party at the respective addresses set forth in Section 7.1.

**7.2. Exhibits.** Exhibits, schedules and annexes referred to in this Agreement and attached hereto are incorporated herein in full by this reference as if each of such exhibits, schedules or annexes were set forth in the body of this Agreement and duly executed by the parties hereto.

**7.3.** Additional Documents and Acts. Each party agrees that it will use all reasonable efforts to take, or cause to be taken, all actions and to do, or cause to be done, all things necessary, proper or advisable, including, but not limited to, the execution of additional documents and instruments, to consummate, make effective and carry out the transactions contemplated by this Agreement.

7.4. Amendment, Modification or Waiver. No amendment, modification or waiver of any condition, provision or term of this Agreement shall be valid or of any effect unless made in writing, signed by the party or parties to be bound or its duly authorized representative and specifying with particularity the nature and extent of such amendment, modification or waiver. Any waiver by any party of a default of another party shall not affect or impair any right arising from any subsequent default.

**7.5. Severable Provisions.** Whenever possible, each provision of this Agreement will be interpreted in such manner as to be effective and valid under applicable law, but if any provision of this Agreement is held to be invalid, illegal or unenforceable under any applicable law or rule in any jurisdiction, such provision will be ineffective only to the extent of such invalidity, illegality, or unenforceability in such jurisdiction, without invalidating the remainder of this Agreement in such jurisdiction or any provision hereof in any other jurisdiction.

**7.6. Entire Agreement.** This Agreement contains the entire understanding of the parties hereto in respect of the transactions contemplated hereby and supersedes all prior agreements and understandings between the parties with respect to such subject matter.

7.7. Captions, Headings, Titles or References to Gender. All captions, headings

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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or titles in the paragraphs or sections of this Agreement are inserted for convenience of reference only and shall not constitute a part of this Agreement or as a limitation of the scope of the particular paragraphs or sections to which they apply. Where appropriate, the masculine gender may be read as the feminine gender or the neuter gender, the feminine gender may be read as the masculine gender or the neuter gender and the neuter gender may be read as the masculine gender or the feminine gender.

**7.8.** Counterparts. This Agreement may be executed in two (2) or more counterparts, each of which shall be considered one and the same Agreement and shall become effective when one or more counterparts have been signed by each of the parties and delivered to the other parties.

IN WITNESS WHEREOF, the parties have executed this Agreement on the date first written above.

AQUA INNOVATIONS, INC.

Kali II +

Dick Disrud its COO

**OXYGENATOR WATER TECHNOLOGIES, INC.** 

Jeff Brink its CEO

EXHIBIT "A"

25

## DESCRIPTION OF LICENSOR PATENTS AND PATENTS PENDING

## United States Patent Number: US 6,689,262 B2 Date of Patent: February 10, 2004 Name: Microbubbles of Oxygen Application Number: 10/372,017

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen.

United States Patent Number: US 7,396,441 B2 Publication Date: July 8, 2008 Name: Flow-Thru Oxygenator Application Number: 10/732,326

An oxygen emitter which is an electrolytic cell. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersatured in oxygen. A flow-through model for oxygenating flowing water. The use of supersaturated water for enhancing the growth of plants. Method of applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture. The treatment of waste water by raising the dissolved oxygen with the use of oxygen emitter.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 383 of 1320

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Page 1 of 1
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Filing or 371 (c) Date:	01-31-2008	Status:	Patented Case
Application Type:	Utility	Status Date:	02-10-2010
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Class / Subclass:	210/748	Patent Number:	7,670,495
First Named Inventor:	James Andrew Senkiw , Minneapolis, MN (US)	Issue Date of Patent:	03-02-2010
	Patent Application I 12/023,431 Select Provide Bibliographic De Bibliographic De Application Number: Filing or 371 (c) Date: Application Type: Examiner Name: Group Art Unit: Confirmation Number: Attorney Docket Number: Class / Subclass: First Named	Patent Application Information Retrieval         Corder Certified Application         12/023,431       FLOW-THROUGH DXYGI         Select Projection Image File Data         Select New Case       Projection         Bibliographic Data       Projection         Application Number:       12/023,431         Filing or 371 (c) Data:       01-31-2008         Application Type:       Utility         Examiner Name:       ALLEN, CAMERON J         Group Art Unit:       1797         Confirmation Number:       7381         Attorney Docket Number:       4056.02US03         Class / Subclass:       210/748         First Named       James Andrew Senkiw ,	Patent Application Information Retrieval         Confer Certified Application As Filed Order Certified File         12/033,431       FLOW-THROUGH DXYGENATOR         Select Projections Transaction Timage File Patent Term Contexcay Data         Select Point Data       Contexcay Waspeer         Application Number:       12/023,431       Customer Number:         Bibliographic Data       Customer Number:         Application Number:       12/023,431       Customer Number:         Filing or 371 (c) Date:       01-31-2008       Status:         Application Type:       Utility       Status Date:         Examiner Name:       ALLEN, CAMERON J       Location:         Group Art Unit:       1797       Location Date:         Confirmation Number:       7381       Earliest Publication No:         Attorney Docket Number:       4056.02US03       Earliest Publication Date:         Class / Subclass:       210/748       Patent Number:         First Named       James Andrew Senkiw ,       Location to the of Patent in the patent of

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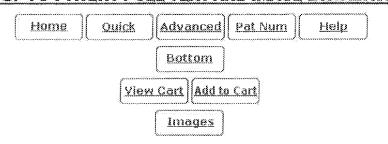
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THIS PATENT ISSUED 3/2/2010. In DISCUSSIONS WITH PAUL HAMN, IT GIVES US BREAD COVERAGE.

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#### **JA381**

Page 1 of 12



# USPTO PATENT FULL-TEXT AND IMAGE DATABASE

United States Patent Senkiw 7,670,495 March 2, 2010

(1 of 1)

Flow-through oxygenator

#### Abstract

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flowthrough model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an oxygen emitter is disclosed.

Inventors: Senkiw; James Andrew (Minneapolis, MN) Assignee: Oxygenator Water Technologies, Inc. (Minnetonka, MN) Appl. No.: 12/023,431 Filed: January 31, 2008

#### **Related U.S. Patent Documents**

<b>Application</b> Number	<b>Filing Date</b>	Patent Number	<b>Issue Date</b>
10732326	Dec., 2003	7396441	
10372017	Feb., 2004	6689262	
60358534	Feb., 2002		

Current U.S. Class:	204/232; 204/245; 205/628; 210/243; 210/600
Current International Class:	C02F 1/48 (20060101); C02F 1/00 (20060101); C25B
1977 - 19 - 9 - 6° 6° P	1/02 (20060101); C25B 1/04 (20060101)
Field of Search:	210/748,600,243 204/278,242,243,275.1,232,286.1,554,660 205/633-638

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JA382

Page 2 of 12

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Primary Examiner: Griffin; Walter D Assistant Examiner: Allen; Cameron J Attorney, Agent or Firm: Patterson, Thuente, Skaar & Christensen, P.A.

## Parent Case Text

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JA383

Page 3 of 12

## **RELATED APPLICATIONS**

This application is a division of application Ser. No. 10/732,326 filed Dec. 10, 2003, which in turn is a continuation-in-part of application Ser. No. 10/372,017, filed Feb. 21, 2003, now U.S. Pat. No. 6,689,262, which claims the benefit of U.S. Provisional Application No. 60/358,534, filed Feb. 22, 2002, each of which is hereby fully incorporated herein by reference.

# Claims

The invention claimed is:

1. A method for treating waste water comprising; providing a flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other, placing the emitter within a conduit; and passing waste water through the conduit.

2. An emitter for electrolytic generation of microbubbles of oxygen in an aqueous medium comprising: an anode separated at a critical distance from a cathode, a nonconductive spacer maintaining the separation of the anode and cathode, the nonconductive spacer having a spacer thickness between 0.005 to 0.050 inches such that the critical distance is less than 0.060 inches and a power source all in electrical communication with each other, wherein the critical distance results in the formation of oxygen bubbles having a bubble diameter less than 0.0006 inches, said oxygen bubbles being incapable of breading the surface tension of the aqueous medium such that said aqueous medium is supersaturated with oxygen.

3. The emitter of claim 2, wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

4. The emitter of claim 2, wherein the anode is platinum and iridium oxide on a support.

5. The emitter of claim 2, wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

6. The emitter of claim 2, wherein the critical distance is 0.005 to 0.060 inches.

7. The emitter of claim 2, comprising a plurality of anodes separated at the critical distance from a plurality of cathodes.

8. A method for oxygenating a non-native habitat for temporarily keeping aquatic animals, comprising: inserting the emitter of claim 2 into the aqueous medium, the non-native habitat comprising an aquarium, a bait bucket or a live well.

9. A method for lowering the biologic oxygen demand of polluted water comprising: passing the polluted water through a vessel containing the emitter of claim 2.

10. A supersaturated aqueous product formed with the emitter of claim 2, the supersaturated aqueous product having an approximately neutral pH.

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 387 of 1320

#### United States Patent: 7670495

Page 4 of 12

11. The emitter of claim 2, further comprising a timer control.

12. The emitter of claim 2, wherein the anode and cathode are arranged such that the emitter assumes a funnel or pyramidal shaped emitter.

Description

## FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

## BACKGROUND OF THE INVENTION

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is described as having an increased biological oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. U.S. Pat. No. 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. U.S. Pat. No. 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

TABLE-US-00001 AT THE CATHODE: 4H.sub.2O + 4e.sup.- .fwdarw. 4OH.sup.- + 2H.sub.2 AT THE ANODE: 2H.sub.2O .fwdarw. O.sub.2 + 4H.sup.+ + 4e.sup.- NET REACTION: 6H.sub.2O .fwdarw. 4OH.sup.- + 4H.sup.+ ++ 2H.sub.2 + O.sub.2

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## JA385

Page 5 of 12

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. U.S. Pat. No. 5,982,609 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U.S. Pat. No. 4,252,856 comprises vacuum deposited iridium oxide.

Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

## SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

JA386

Page 6 of 12

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack. Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.

This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the O.sub.2 emitter of the invention.

FIG. 2 is an assembled device.

FIG. 3 is a diagram of the electronic controls of the O.sub.2 emitter.

FIG. 4 shows a funnel or pyramid variation of the O.sub.2 emitter.

FIG. 5 shows a multilayer sandwich O.sub.2 emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-through applications. FIG. 7A is a cross section showing arrangement of three plate electrodes. FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

## DETAILED DESCRIPTION OF THE INVENTION

Definitions

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O.sub.2 emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O.sub.2. In the special dimensions of the invention, as explained in more detail in the following examples, O.sub.2 forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H.sub.2 formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

## EXAMPLE 1

## Oxygen Emitter

As shown in FIG. 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, Ohio). The cathode 2 is a (fraction (1/16)) inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2

http://patft1.uspto.gov/netacgi/nph-Parser?Sect1=PTO1&Sect2=HITOFF&d=PALL&p=1&... 3/2/2010

Page 8 of 12

shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon.RTM., polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an O.sub.2 emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. FIG. 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

## EXAMPLE 2

## Measurement of O.sub.2 Bubbles

Attempts were made to measure the diameter of the O.sub.2 bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.

Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed O.sub.2 bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

## EXAMPLE 3

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Page 9 of 12

Other Models of Oxygen Emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H.sub.2) arise at the center of the emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. FIG. 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

 TABLE-US-00002 TABLE I Emitter Model Gallons Volts Amps Max. Ave Watts Bait keeper 5 6 0.090
 0.060 0.36 Livewell 32 12 0.180 0.120 1.44 OEM 2 inch 10 12 0.210 0.120 1.44 Bait store 70 12 0.180

 0.180 2.16 Double cycle 2 12 0.180 0.180 2.16 OEM 3 inch 50 12 0.500 0.265 3.48 OEM 4 inch 80 12
 0.980 0.410 4.92 Water pail 2 24 1.200 1.200 28.80 Plate 250 12 5.000 2.500 30.00

**EXAMPLE 4** 

Multilayer Sandwich O.sub.2 Emitter

An O.sub.2 emitter was made in a multilayer sandwich embodiment. (FIG. 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

TABLE-US-00003 cathode screen 0.045 inches thick nylon spacer 0.053 inches thick anode grid 0.035 inches thick nylon spacer 0.053 inches thick cathode screen 0.045 inches thick,

for an overall emitter thickness of 0.231 inches thick inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, cathode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

**EXAMPLE 5** 

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Page 10 of 12

Effect of Superoxygenated Water on the Growth of Plants

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. U.S. Pat. No. 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air. Such a method has high energy requirements and is noisy. Furthermore, while suitable for self-contained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may become saturated with oxygen, but it is unlikely that the water is superoxygenated.

A. Superoxygenated Water in Hydroponic Culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 1/2 gallon water reservoir for the Control plant was eroated with and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to 165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

B. Superoxygenated Water in Field Culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in one-inch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One

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Page 11 of 12

half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

TABLE-US-00004 TABLE II Control, grams Test, grams tomatoes from tomatoes from eight plants/ seven plants/ Week of: cumulative total cumulative total July 27 240 400 August 3 180 420 2910 3310 August 10 905 1325 1830 5140 August 17 410 1735 2590 7730 August 24 3300 5035 2470 10200 August 31 4150 9175 1580 11780 September 15 not weighed 3710 15490 Final Harvest 6435 15620 8895 24385 September 24

The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

FIG. 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

## **EXAMPLE 6**

Flow-Through Emitter for Agricultural Use

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In FIG. 7(A), the oxygenation chamber is comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120.degree. angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. FIG. 7(B) shows a plan view of the oxygenation chamber with stabilizing hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art can readily fabricate any of the emitters shown in FIG. 4 or 5, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2.degree. C. but the flowing water cooled slightly to 11 or 11.5.degree. C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

TABLE-US-00005 TABLE III ACTIVE DO OF\* ELECTRODE CURRENT, FLOW RATE SAMPLE AT MODEL AREA, SQ.IN. VOLTAGE AMPS. GAL/MINUTE ONE MINUTE 2-Inch "T" 2 28.3 0.72 12 N/A 3-inch "T" 3 28.3 1.75 12 N/A 2-plate Tube 20 28.3 9.1 12 8.4 3-Plate tube 30 28.3 12.8 12 9.6

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JA392

Page 12 of 12

\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The oneminute time point shows the rapid increase in oxygenation.

The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

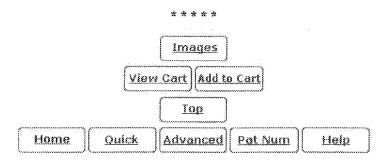
It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

EXAMPLE 7

Treatment of Waste Water

Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in FIG. 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.



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JA393



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 UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.usplo.gov

 APPLICATION NO.
 ISSUE DATE
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 ATTORNEY DOCKET NO.
 CONFIRMATION NO.

 10/732,326
 07/08/2008
 7396441
 4056.02US01
 7020

24113 7590 06/18/2008 PATTERSON, THUENTE, SKAAR & CHRISTENSEN, P.A. 4800 IDS CENTER 80 SOUTH 8TH STREET MINNEAPOLIS, MN 55402-2100

# **ISSUE NOTIFICATION**

The projected patent number and issue date are specified above.

# Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)

(application filed on or after May 29, 2000)

The Patent Term Adjustment is 7 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

James Andrew Senkiw, Minneapolis, MN;

IR103 (Rev. 11/05)

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								(Signature)
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	APPLICATION NO.	FILING DATE		FIRST NAMED INVEN	TOR	ATTORNEY	DOCKET NO.	CONFIRMATION NO.
	10/732,326	12/10/2003		James Andrew Sen	kiw		<del>102USI</del> 56.02USC	7020
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-	nonprovisional	YES	\$720	\$300	\$0		\$1020	07/23/2008
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Electronic Patent Application Fee Transmittal					
Application Number:	10	732326			
Filing Date:	10-Dec-2003				
Title of Invention:	FL	.OW-THROUGH C	DXYGENATO	R	
First Named Inventor/Applicant Name:	Ja	mes Andrew Senk	<b>liw</b>		
Filer:         J. Paul Haun/Valerie Mitchell			Mitchell		
Attorney Docket Number:	40	56.02US01			
Filed as Small Entity					
Utility Filing Fees					
Description		Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:					
Pages:					
Claims:					
Miscellaneous-Filing:					
Petition:					
Patent-Appeals-and-Interference:					
Post-Allowance-and-Post-Issuance:					
Utility Appl issue fee		2501	1	720	720
Publ. Fee- early, voluntary, or normal		1504	1	300	300

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 399 of 1320

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Extension-of-Time:				
Miscellaneous:				
Printed copy of patent - no color	8001	10	3	30
Total in USD (\$)				1050

Electronic Ac	knowledgement Receipt
EFS ID:	3335034
Application Number:	10732326
International Application Number:	
Confirmation Number:	7020
Title of Invention:	FLOW-THROUGH OXYGENATOR
First Named Inventor/Applicant Name:	James Andrew Senkiw
Customer Number:	24113
Filer:	J. Paul Haun/Valerie Mitchell
Filer Authorized By:	J. Paul Haun
Attorney Docket Number:	4056.02US01
Receipt Date:	21-MAY-2008
Filing Date:	10-DEC-2003
Time Stamp:	11:55:17
Application Type:	Utility under 35 USC 111(a)

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Payment was successfully received in RAM	\$1050			
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# NOTICE OF ALLOWANCE AND FEE(S) DUE

24113 7590 04/23/2008 PATTERSON, THUENTE, SKAAR & CHRISTENSEN, P.A. 4800 IDS CENTER 80 SOUTH 8TH STREET MINNEAPOLIS, MN 55402-2100

EXAMINER				
ZHENG, LOIS L				
ART UNIT	PAPER NUMBER			
1793				

DATE MAILED: 04/23/2008

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.	
10/732,326	12/10/2003	James Andrew Senkiw	AQL.002US1	7020	
TTT F OF BREATHON, FLOW THROUGH OWNGENATOR					

TITLE OF INVENTION: FLOW-THROUGH OXYGENATOR

APPLN. TYPE	SMALL ENTITY	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	YES	\$720	\$300	\$0	\$1020	07/23/2008

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. <u>PROSECUTION ON THE MERITS IS CLOSED</u>. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN <u>THREE MONTHS</u> FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. <u>THIS STATUTORY PERIOD CANNOT BE EXTENDED</u>. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

#### HOW TO REPLY TO THIS NOTICE:

I. Review the SMALL ENTITY status shown above.

If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status:	If the SMALL ENTITY is shown as NO:
A. If the status is the same, pay the TOTAL FEE(S) DUE shown above.	A. Pay TOTAL FEE(S) DUE shown above, or
B. If the status above is to be removed, check box 5b on Part B - Fee(s) Transmittal and pay the PUBLICATION FEE (if required) and twice the amount of the ISSUE FEE shown above, or	B. If applicant claimed SMALL ENTITY status before, or is now claiming SMALL ENTITY status, check box 5a on Part B - Fee(s) Transmittal and pay the PUBLICATION FEE (if required) and 1/2 the ISSUE FEE shown above.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

Page 1 of 3

PTOL-85 (Rev. 08/07) Approved for use through 08/31/2010.

JA400

## PART B - FEE(S) TRANSMITTAL

INSTRUCTIONS: This form			or <u>Fax</u>	Ale	). Box 1450 xandria, Virginia 2 1)-273-2885	2313-1450	
indicated unless corrected bel maintenance fee notifications.	n should be used for spondence includin low or directed oth	or transmitting the ISS g the Patent, advance of erwise in Block 1, by (	UE FEE and PUBLIC orders and notification (a) specifying a new c	CATI of m corresj	ON FEE (if required). E naintenance fees will be pondence address; and/or	Blocks 1 through 5 sl mailed to the current (b) indicating a sepa	nould be completed where correspondence address as rate "FEE ADDRESS" for
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MINNEAPOLIS, MI	N 55402-2100						(Depositor's name)
							(Signature)
							(Date)
APPLICATION NO.	FILING DATE		FIRST NAMED INVEN	ITOR	ATTO	RNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003		James Andrew Sen	kiw		AQL.002US1	7020
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nonprovisional	YES	\$720	\$300		\$0	\$1020	07/23/2008
EXAMINER		ART UNIT	CLASS-SUBCLASS	S			
ZHENG, LOIS 1. Change of correspondence a		1793	204-242000		atent front page, list		
CFR 1.363). Change of corresponder Address form PTO/SB/122 PTO/SB/47; Rev 03-02 or Number is required. 3. ASSIGNEE NAME AND R	n (or "Fee Address" more recent) attach	Indication form ed. Use of a Customer	or agents OR, alte (2) the name of a registered attorney 2 registered paten listed, no name wi	rnativ single y or a t attor ill be p	e firm (having as a memb gent) and the names of u neys or agents. If no nam printed.	er a 2 p to	
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5. Change in Entity Status (fi	ALL ENTITY statu	s. See 37 CFR 1.27.	b. Applicant is no	o long	er claiming SMALL EN	FITY status. See 37 CI	FR 1.27(g)(2).
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PTOL-85 (Rev. 08/07) Approved for use through 08/31/2010.

OMB 0651-0033

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

JA401

	ited States Pate	ENT AND TRADEMARK OFFICE	UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F O. Box 1450 Alexandria, Virginia 223 www.usplo.gov	OR PATENTS
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003	James Andrew Senkiw	AQL.002US1	7020
24113 7	590 04/23/2008		EXAN	IINER
PATTERSON, 7	HUENTE, SKAAR	& CHRISTENSEN, P.A.	ZHENG	, LOIS L
4800 IDS CENTE			ART UNIT	PAPER NUMBER
80 SOUTH 8TH S MINNEAPOLIS,			1793 DATE MAILED: 04/23/200	8

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (application filed on or after May 29, 2000)

The Patent Term Adjustment to date is 7 day(s). If the issue fee is paid on the date that is three months after the mailing date of this notice and the patent issues on the Tuesday before the date that is 28 weeks (six and a half months) after the mailing date of this notice, the Patent Term Adjustment will be 7 day(s).

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

Page 3 of 3

**JA402** 

	Application No.	Applicant(s)
	10/732,326	SENKIW, JAMES ANDREW
Notice of Allowability	Examiner	Art Unit
	LOIS ZHENG	1793
The MAILING DATE of this communication appe All claims being allowable, PROSECUTION ON THE MERITS IS rerewith (or previously mailed), a Notice of Allowance (PTOL-85) INTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RI of the Office or upon petition by the applicant. See 37 CFR 1.313	(OR REMAINS) CLOSED in or other appropriate commun IGHTS. This application is su	this application. If not included nication will be mailed in due course. <b>THIS</b>
. X This communication is responsive to <u>amendment after fina</u>	<u>l filed 3 March 2008</u> .	
. 🔀 The allowed claim(s) is/are <u>1-4, 9, 13, 15 and 17-26</u> .		
<ul> <li>Acknowledgment is made of a claim for foreign priority ur</li> <li>a) All b) Some* c) None of the: <ol> <li>Certified copies of the priority documents have</li> <li>Certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Copies of the certified copies of the priority documents have</li> <li>Certified copies not received:</li> </ol> </li> <li>Applicant has THREE MONTHS FROM THE "MAILING DATE" noted below. Failure to timely comply will result in ABANDONN</li> </ul>	<ul> <li>been received.</li> <li>been received in Application cuments have been received</li> <li>of this communication to file a</li> </ul>	No in this national stage application from the
<ul> <li>THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.</li> <li>A SUBSTITUTE OATH OR DECLARATION must be subm INFORMAL PATENT APPLICATION (PTO-152) which give</li> <li>CORRECTED DRAWINGS ( as "replacement sheets") mus (a) ☐ including changes required by the Notice of Draftspers</li> </ul>	es reason(s) why the oath or o at be submitted. son's Patent Drawing Review	declaration is deficient.
<ol> <li>⇒ hereto or 2)</li> <li>⇒ to Paper No./Mail Date</li> <li>⇒ including changes required by the attached Examiner's Paper No./Mail Date</li> </ol>		in the Office action of
Identifying indicia such as the application number (see 37 CFR 1 each sheet. Replacement sheet(s) should be labeled as such in t		
DEPOSIT OF and/or INFORMATION about the depo attached Examiner's comment regarding REQUIREMENT		
<ul> <li>Attachment(s)</li> <li>I. ☐ Notice of References Cited (PTO-892)</li> <li>2. ☐ Notice of Draftperson's Patent Drawing Review (PTO-948)</li> <li>3. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date <u>7/19/2004,2/18/08</u></li> <li>I. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material</li> </ul>	6.	<i>I</i> ail Date Amendment/Comment Statement of Reasons for Allowance
J.S. Patent and Trademark Office PTOL-37 (Rev. 08-06) No	otice of Allowability	Part of Paper No./Mail Date 200803

Application/Control Number: 10/732,326 Art Unit: 1793 Page 2

# DETAILED ACTION

# Status of Claims

1. Claims 1 and 25-26 are amended in view of applicants response filed 3 March

2009. Claims 14 and 16 are canceled in view of applicant's response. Claims 5-8 and 10-12 remain withdrawn from consideration. Therefore, claims 1-4, 9, 13, 15 and 17-26 are currently under examination.

# Specification

2. The amendments to specification are entered and recorded.

# Drawing

3. The drawings were received on 3 March 2008. These drawings are acceptable.

# Information Disclosure Statement

4. The information disclosure statement (IDS) submitted on 18 February 2008 was filed after the mailing date of the Final Rejection on 1 November 2007. The submission is in compliance with the provisions of 37 CFR 1.97. Accordingly, the information disclosure statement is being considered by the examiner.

## Status of Previous Rejections

5. All previous rejections are withdrawn in view of applicant's claim amendments filed 3 March 2008.

# Allowance

6. Claims 1-4, 9, 13, 15 and 17-26 are allowed.

7. The following is a statement of reasons for the indication of allowable subject matter: The prior art of record does not teach or fairly suggest, either alone or in

JA404

Application/Control Number: 10/732,326 Art Unit: 1793 Page 3

combination, the claimed flow through oxygenator comprising three matched sets of anodes and cathodes attached to stabilizing hardware in adjacent relation such that each matched set resides at a 120° angle to the adjacent matched sets.

This application is in condition for allowance except for the presence of claims 5 8 and 10-12 directed to invention non-elected without traverse. Accordingly, claims 5-8
 and 10-12 have been cancelled.

#### Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to LOIS ZHENG whose telephone number is (571)272-1248. The examiner can normally be reached on 8:30am - 5:00pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Roy King can be reached on (571) 272-1244. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

JA405

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 408 of 1320

Application/Control Number: 10/732,326 Art Unit: 1793 Page 4

/Roy King/ Supervisory Patent Examiner, Art Unit 1793

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 409 of 1320

Application Number	Application/Control No.	Applicant(s)/Patent un Reexamination	nder	
	10/732,326	SENKIW, JAMES ANDREV		
	Examiner	Art Unit		
	LOIS ZHENG	1793		

U.S. Patent and Trademark Office

Part of Paper No. 20080312

Issue Classification	Application/Control No. 10/732,326	Applicant(s)/Patent under Reexamination SENKIW, JAMES ANDREW
	Examiner LOIS ZHENG	Art Unit 1793

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U.S. Patent and Trademark Office

Part of Paper No. 20080312

**JA408** 

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 411 of 1320

	Search Notes		Application/Control No.	Applicant(s)/Pate Reexamination		
				10/732,326 Examiner	SENKIW, JAME	S ANDRE
				LOIS ZHENG	1793	
	SEAR	CHED			RCH NOTES SEARCH STRATEG	Y)
Class	Subclass	Date	Examiner		DATE	EXMR
204	232	3/10/2008	LLZ	Inventorship search	3/12/2008	LLZ
204	242	3/10/2008	LLZ			
204	275.1	3/10/2008	LLZ			
204	278	3/10/2008	LLZ	Updated EAST search	3/10/2008	LLZ
204	286.1	3/10/2008	LLZ			
204	554	3/10/2008	LLZ			
204	660	3/10/2008	LLZ			
205	633	3/10/2008	LLZ			
205	742	3/10/2008	LLZ			
210	243	3/10/2008	LLZ			
210	748	3/10/2008	LLZ			
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Class	Subclass	Date	Examiner			
204	278	3/10/2008	LLZ			
205	633	3/10/2008	LLZ			
210	243	3/10/2008	LLZ			

JA409

EAST Search History

## EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
S40	1	(204/554-573,660- 674).ccls. and (anode cathode electrode) with angle with "120"	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 14:36
S41	1	(210/243,748).ccls. and (anode cathode electrode) with angle with "120"	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 14:37
S42	4	(210/243,748).ccls. and (anode cathode electrode) with angle with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 14:38
S43	3	(204/554-573,660- 674).ccls. and (anode cathode electrode) with angle with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 15:24
S44	7	"210".clas. and (anode cathode electrode) with angle with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 15:25
S45	115	(204/554-573,660- 674).ccls. and (anode cathode electrode) with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 15:26
S47	174	S46 not S44	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 15:58

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S46	181	"210".clas. and (anode cathode electrode) with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/07 15:58
S53	0	("204" "205").clas. and (anode cathode electrode) with ". degree."	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 09:59
S52	0	"210".clas. and (anode cathode electrode) with ". degree."	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 09:59
S51	0	"210".clas. and (anode cathode electrode) with angle with ".degree."	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 09:59
S54	4479	("204" "205").clas. and (anode cathode electrode) with degree	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 10:00
S55	1537	("204" "205").clas. and (anode cathode electrode) with degree and (anode cathode electrode) with (conduit pipe cylinder channel)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 10:01
S57	1161	S55 not (temperature with degree degree near2 (C F))	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 10:02
S56	1188	S55 not (temperature with degree)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2008/03/10 10:02

EAST Search History

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#### PATENT APPLICATION

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Senkiw

Attorney Docket No.: 4056.02US01

Application No.: 10/732,326

Filed: December 10, 2003

Examiner: Lois L. Zheng Group Art Unit: 1793

Confirmation No.: 7020

For: FLOW-THROUGH OXYGENATOR

#### AMENDMENT AFTER FINAL

Mail Stop AF Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

#### **INTRODUCTORY COMMENTS**

In response to the Final Office Action of November 1, 2007, and in accordance with the automatic extension of time for response provided by 37 CFR § 1.136(a), amendment to the above-identified patent application is requested.

The present amendment comprises the following sections:

- A. Amendments to the Specification
- B. Amendments to the Claims
- C. Amendments to the Drawings

OK TO ENTER: /LZ/

D. Remarks

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 416 of 1320

	Sub	stitute for form 1	449/PTO			Comple	ete if Known				
				Application Numbe	er	10/73	2,326				
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LZ		WO 99/39561		08-12-1999			Mazzei				
LZ		WO 03/07250	07 A1	09-04-2003	3		Senkiw				
EXAMINER SIGNATURE		/Lois. L Zheng/		DATE	DERED		3/17/08				
*EXAMINER: Ini copy of this form v <sup>1</sup> Applicant's uniqu by the two-letter or document. <sup>5</sup> Kind o language Translati This collection of i an application. Con the completed app suggestions for rec	with next co be citation d ode (WIPO of document on is attach information nfidentiality lication for lucing this b	mmunication to applicant. esignation number (optiona Standard ST.3). <sup>4</sup> For Japan by the appropriate symbols ed. is required by 37 CFR 1.97 v is governed by 35 U.S.C. n to the USPTO. Time will urden, should be sent to the	1). <sup>2</sup> See Kinds Codes of USP ese patent documents, the inc as indicated on the document and 1.98. The information i 122 and 37 CFR 1.14. This c vary depending upon the ind	with MPEP 609. Draw lim TO Patent Documents at ww dication of the year of the rei nt under WIPO Standard ST. 's required to obtain or retain ollection is estimated to take tividual case. Any comments U.S. Patent and Trademark (	e through w.uspto.g ign of the . 16 if pos a benefit 2 hours to s on the ar Office, U.	gov or MP Emperor a ssible. <sup>6</sup> Ap by the put to complet mount of t S. Departi	f not in conformance and not considered. In 'EP 901.04. <sup>3</sup> Enter Office that issued the dor must precede the serial number of the paten plicant is to place a check mark here if Eng blic which is to file (and by the USPTO to p e, including gathering, preparing, and subm ime you require to complete this form and/c ment of Commerce, Washington, DC 20231 undria, VA 22313-1450.	cument, t lish process) itting pr			

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	e for form 1449/PTO				Complete If Known
				Application Number	10/732,326
			CLOSURE	Filing Date	December 10, 2003
STA	TEMENT E	BY A	PPLICANT	First Named Inventor	James Andrew Senkiw
	(Use as many she			Art Unit	
	less as many suc	via 68 /i	ecessaryj	Examiner Name	
Sheet	2	of	2	Attorney Docket Number	AQI.002US1

Examiner Initials*	Cite No. <sup>1</sup>	NON PATENT LITERATURE DOCUMENTS Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Т
,06		Effect of Oxygenated Water on the Growth and Biomass Development of Seedless Cucumbers and Tomato Seedlings under Greenhouse Conditions: Mohyuddin Mirza et al. www.seair.ca 2003	

 Examiner
 Date
 (1/21/05)

 "EXAMINER: Initial if reference considered, whether or not dilation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.
 Date
 (1/21/05)

 "Applicant's unique diation designation number (optional). 2 Applicant is to place a check mark here if English language Transistion is attached.
 This collection of information is required by 37 CFR 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an applicatiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is diverted to take 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of file, P.O. Box 1450, Alaxandria, VA 22313.1450. Do NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313.1450.

If you need assistance in completing the form, call 1-800-PTO-9199 (1-800-786-9199) and select option 2.

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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#### PATENT APPLICATION

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Senkiw

Attorney Docket No.: 4056.02US01

Application No.: 10/732,326

Filed: December 10, 2003

Group Art Unit: 1793

Examiner: Lois L. Zheng

Confirmation No.: 7020

For: FLOW-THROUGH OXYGENATOR

#### AMENDMENT AFTER FINAL

Mail Stop AF Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

#### **INTRODUCTORY COMMENTS**

In response to the Final Office Action of November 1, 2007, and in accordance with the automatic extension of time for response provided by 37 CFR § 1.136(a), amendment to the above-identified patent application is requested.

The present amendment comprises the following sections:

- A. Amendments to the Specification
- B. Amendments to the Claims
- C. Amendments to the Drawings
- D. Remarks

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### AMENDMENTS TO THE SPECIFICATION

In the Specification

Please substitute the following amended paragraph(s) and/or section(s) (deleted matter is

shown by strikethrough and added matter is shown by underlining):

Page 5, lines 5-23,

#### DESCRIPTION OF THE DRAWINGS

FIG. 1A is a plan view of an [[the]] O<sub>2</sub> emitter of the invention.

FIG. 1B is a section view of the O<sub>2</sub> emitter of Figure 1A taken at line 1B-1B of FIG. 1A.

FIG. 2<u>A</u> is a plan view of an assembled  $O_2$  emitting device.

FIG. 2B is a perspective view of the assembled 02 emitting device of FIG. 2A.

FIG. 3 is a diagram of the electronic controls of the  $O_2$  emitter.

FIG. 4 shows a funnel or pyramid variation of the O<sub>2</sub> emitter.

FIG. 5 shows a multilayer sandwich  $O_2$  emitter.

FIG. 6 shows the yield of tomato plants watered with superoxygenated water.

FIG. 7 shows an oxygenation chamber suitable for flow-through applications.

FIG. 7A is a cross section showing arrangement of three plate electrodes.

FIG. 7B is a longitudinal section showing the points of connection to the power source.

FIG. 8 is a graph showing the oxygenation of waste water.

Page 7, line 16 – page 8, line 2,

2

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

As shown in FIGS. 1A, 1B, 2A and 2B, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, OH). The cathode 2 is a {fraction (1/16)} inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. FIG. 2A shows a plan view of the assembled device. The O.sub.2 emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon.RTM. polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

3

**JA418** 

#### AMENDMENTS TO THE CLAIMS

A detailed listing of all claims that are, or were, in the present application, irrespective of whether the claim(s) remains under examination in the application are presented below. The claims are presented in ascending order and each includes one status identifier. Those claims not cancelled or withdrawn but amended by the current amendment utilize the following notations for amendment: 1. deleted matter is shown by strikethrough for six or more characters and double brackets for five or less characters; and 2. added matter is shown by underlining.

4

1. (Currently Amended) A flow through oxygenator comprising:

a fluid conduit having a fluid inlet and a fluid outlet fluidly connected with a conduit lumen;

an oxygen emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, the oxygen emitter including a plurality of three matched sets of anodes and cathodes wherein the matched sets of anodes and cathodes are mounted to stabilizing hardware such that the oxygen emitter is positioned within the conduit lumen <u>and each</u> matched set resides at a 120° angle to the adjacent matched sets; and

a power source in electrical communication with the oxygen emitter.

2. (Previously Presented) The flow through oxygenator of claim 1, wherein each anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and each cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

3. (Previously Presented) The flow through oxygenator of claim 1, wherein the anode and cathode within each matched set are separated by a spacer such to maintain a gap of 0.005 to 0.140 inches between the anode and cathode.

4. (Previously Presented) The flow through oxygenator of claim + 3, wherein the gap is 0.045 to 0.060 inches.

5. (Withdrawn) The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

5

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

6. (Withdrawn) A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

9. (Previously Presented) The flow through oxygenator of claim 1 wherein each anode is platinum and iridium oxide on a support and each cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

10. (Withdrawn) A method to increase the oxygen content of flowing water comprising passing flowing water through a conduit comprising the flow-through oxygenator of claim 1.

11. (Withdrawn) The method of claim 11 wherein the flowing water has a temperature of 1 to40 degrees Celsius.

12. (Withdrawn) The method of claim 11 wherein the flowing water becomes supersaturated with oxygen.

13. (Previously Presented) The flow through oxygenator of claim 1, wherein the power source is electrically connected to the stabilizing hardware for powering the plurality of matched sets of anodes and cathodes.

6

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

14. Cancelled.

15. (Previously Presented) The flow through oxygenator of claim 1, wherein the plurality of matched sets of anodes and cathodes are attached to the stabilizing hardware with the anodes proximate a conduit wall and the cathodes proximate a conduit center.

16. Cancelled.

17. (Previously Presented) The flow through oxygenator of claim 1, wherein the plurality of matched sets of anodes and cathodes define plates positioned parallel to a flow axis of the conduit lumen.

18. (Previously Presented) The flow through oxygenator of claim 1, wherein each cathode comprises a mesh screen.

19. (Previously Presented) The flow through oxygenator of claim 1, further comprising: a controller selectively operating the power source, such that the power source supplies power to the plurality of matched sets of anodes and cathodes when the aqueous medium is flowing through the conduit lumen and withholds power when the aqueous medium is not flowing through the conduit lumen.

20. (Previously Presented) The flow through oxygenator of claim 1, wherein the oxygen emitter is sized to generate oxygen sufficient to form a supersaturated aqueous medium.

7

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

21. (Previously Presented) The flow through oxygenator of claim 1, wherein the aqueous medium is water.

22. (Previously Presented) The flow through oxygenator of claim 21, wherein the oxygen emitter is sized to generate oxygen sufficient to form superoxygenated water.

23. (Previously Presented) The flow through oxygenator of claim 1, wherein the fluid conduit is a watering hose.

24. (Previously Presented) The flow through oxygenator of claim 1, wherein the fluid conduit is a hydroponic circulating system.

25. (Currently Amended) A flow through oxygenator comprising:

a watering hose having a hose lumen; and

an oxygen emitter operably mounted within the hose lumen, the oxygen emitter including three matched sets of anodes and cathodes mounted to stablilizing hardware such that each matched set resides at a 120° angle to the adjacent matched sets.

26. (Currently Amended) A flow through oxygenator comprising:
 a hydroponic circulating system having a circulating lumen; and
 an oxygen emitter operably mounted within the circulating lumen, the
 oxygen emitter including three matched sets of anodes and cathodes mounted to
 stablilizing hardware such that each matched set resides at a 120° angle to the
 adjacent matched sets.

8

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### AMENDMENTS TO THE DRAWINGS

Attachment: Qty. 3 Replacement Sheets

Replacement sheets to correct identified deficiencies Figures 6, 7 and 8 are submitted to correct said deficiencies.

#### <u>REMARKS</u>

Claims 1-4, 9 and 13-26 are pending. By this Amendment, claims 14 and 16 are cancelled and claims 1, 25 and 26 are amended. By way of the present amendments to independent claims 1, 25 and 26, Applicant has included the subject matter of former dependent claim 14, which was indicated as being allowable in the Final Office Action mailed November 1, 2007. No new matter is believed introduced by way of the present amendments.

#### **Drawings**

In the Final Office Action mailed November 1, 2007, the drawings were objected to based on a number of identified deficiencies. Applicant respectfully requests said objections be withdrawn.

In response to the objections regarding Figures 1A, 1B, 2A and 2B, Applicant has amended the specification to discuss all of the submitted Figures.

In response to the objections to Figure 6, Applicant submits a replacement Figure 6 removing the additional data points and more accurately representing the data points.

In response to the objection to Figures 7(A) and 7(B), Applicant submits a replacement sheet and amends the specification to overcome said deficiencies.

In response to the objection to Figure 8, Applicant submits replacement Figure 8 as described in the application. In reviewing the original filing submission, it appears that an incorrect Figure was erroneously supplied as Figure 8. Applicant respectfully asserts that replacement Figure 8 is described and inherently supported in the original specification such that new Figure 8 does not constitute new matter.

Applicant has respectfully cancelled claim 16 including the limitation of a side arm flow portion.

**JA425** 

### Priority

Applicant respectfully takes no position concerning the effective filing date of the present application.

#### Claim Rejections 35 USC 102

In the Final Office Action mailed November 1, 2007, claims 1-3, 13, 15 and 17-22 were rejected under 35 USC 102(b) as being anticipated by U.S. Patent Publication No. 2002/0074237 A1 to Takesako. In the Final Office Action mailed November 1, 2007, claims 1-2, 13, 17 and 20-22 were rejected under 35 USC 102(b) as being anticipated by U.S. Patent No. 6,171,469 to Hough. By way of the present amendment to independent claim 1, Applicant has incorporated the previously indicated allowable subject matter of former dependent claim 14. As such, Applicant respectfully requests said rejections be withdrawn.

### Claim Rejections 35 USC 103

In the Final Office Action mailed November 1, 2007, claims 4, 16 and 23-26 were rejected under 35 USC 103(a) as being unpatentable over Takesako. In the Final Office Action mailed November 1, 2007, claim 9 was rejected under 35 USC 103(a) as being unpatentable over Takesako in view of U.S. Patent No. 4,587,001 to Cairns et al. In the Final Office Action mailed November 1, 2007, claims 3-4, 16, 18-19 and 23-26 were rejected under 35 USC 103(a) as being unpatentable over Hough, and further in view of Takesako. In the Final Office Action mailed November 1, 2007, claim 9 was rejected under 35 USC 103(a) as being unpatentable over Hough, and further in view of Takesako. In the Final Office Action mailed November 1, 2007, claim 9 was rejected under 35 USC 103(a) as being unpatentable over Hough, in view of Cairns. By way of the present amendment to independent claims 1, 25 and 26, Applicant has incorporated the previously indicated allowable subject matter of former dependent claim 14. As such, Applicant respectfully requests said rejections be withdrawn.

**JA426** 

### Double Patenting

In the Final Office Action mailed November 1, 2007, claims 1-4, 9, 13, 15 and 18-22 were rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1, 6, 9 and 13-14 of U.S. Patent No. 6,689,262 in view of Takesako. By way of the present amendment to independent claim 1, Applicant has incorporated the previously indicated allowable subject matter of former dependent claim 14. As such, Applicant respectfully requests said rejections be withdrawn.

In view of the foregoing, it is submitted that this application is in condition for allowance. Favorable consideration and prompt allowance of the application are respectfully requested.

The Examiner is invited to telephone the undersigned if the Examiner believes it would be useful to advance prosecution.

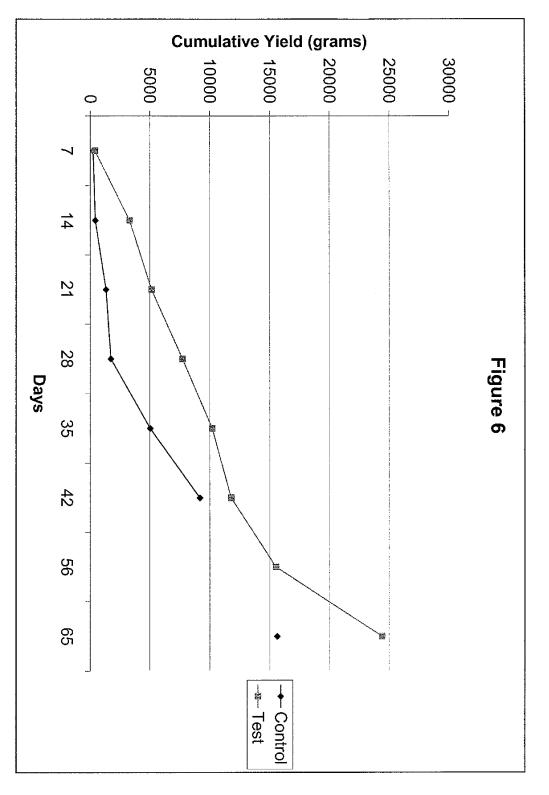
Respectfully submitted J. Paul Haun Registration No. 53,003

Customer No. 24113 Patterson, Thuente, Skaar & Christensen, P.A. 4800 IDS Center 80 South 8th Street Minneapolis, Minnesota 55402-2100 Telephone: (612) 349-3009

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

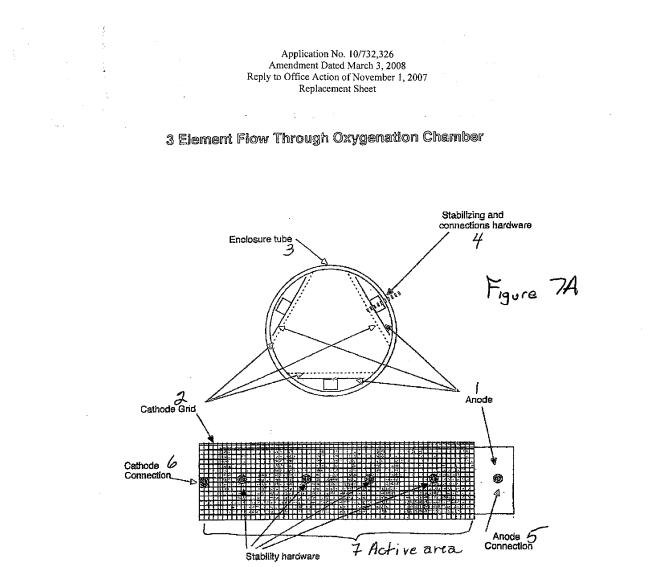
# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 430 of 1320

Application No. 10/732,326 Amendment Dated March 3, 2008 Reply to Office Action of November 1, 2007 Replacement Sheet



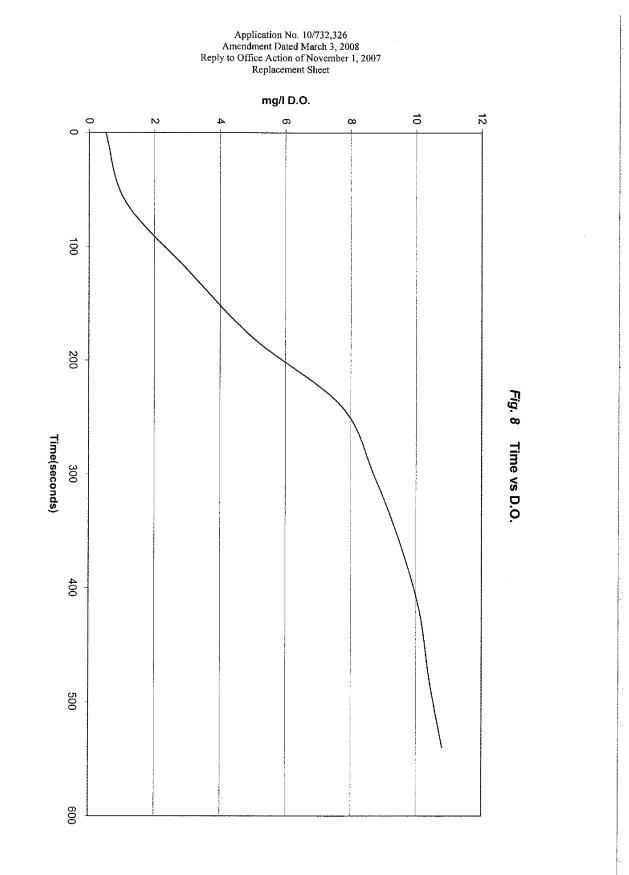
JA428

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 431 of 1320



Depending on requirements tube can contain 1 2 3 4 or more elements.

Figure 7B



OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## PATENT APPLICATION

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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Senkiw

Application No.: 10/732,326

Filed: December 10, 2003

Attorney Docket No.: 4056.02US01

Confirmation No.: 7020

Examiner: Lois L. Zheng

Group Art Unit: 1793

For: FLOW-THROUGH OXYGENATOR

# PETITION FOR EXTENSION OF PERIOD FOR RESPONSE UNDER 37 CFR § 1.136(a)

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Pursuant to 37 CFR § 1.136(a), an extension of time of one (1) month (from February 1, 2008 to March 3, 2008, March 1, 2008 falling on a Saturday) within which to respond to the Office Action dated November 1, 2007 is requested. Please charge the \$60.00 one month extension fee to Deposit Account No. 16-0631. Applicant is entitled to small entity status in accordance with 37 CFR 1.27. The Commissioner is authorized to charge to Deposit Account No. 16-0631 any underpayments, overpayments or additionally required fees.

Respectfully submitte J. Raul Haun Registration No. 53,003

Customer No. 24113 Patterson, Thuente, Skaar & Christensen, P.A. 4800 IDS Center 80 South 8th Street Minneapolis, Minnesota 55402-2100 Telephone: (612) 394-3009

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Electronic Patent Application Fee Transmittal						
Application Number:	10732326					
Filing Date:	10	-Dec-2003				
Title of Invention:	Flow-through oxygenator					
First Named Inventor/Applicant Name:	Ja	mes Andrew Senk	iw			
Filer:	J. Paul Haun/Allison Goette					
Attorney Docket Number:	AQI.002US1					
Filed as Small Entity						
Utility Filing Fees						
Description		Fee Code	Quantity	Amount	Sub-Total in USD(\$)	
Basic Filing:						
Pages:						
Claims:						
Miscellaneous-Filing:						
Petition:						
Patent-Appeals-and-Interference:						
Post-Allowance-and-Post-Issuance:						
Extension-of-Time:						
Extension - 1 month with \$0 paid		2251	1	60	60	

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 435 of 1320

Description	Amount	Sub-Total in USD(\$)		
Miscellaneous:				
	Total in USD (\$)			60

Electronic Ac	Electronic Acknowledgement Receipt					
EFS ID:	2942752					
Application Number:	10732326					
International Application Number:						
Confirmation Number:	7020					
Title of Invention:	Flow-through oxygenator					
First Named Inventor/Applicant Name:	James Andrew Senkiw					
Customer Number:	24113					
Filer:	J. Paul Haun					
Filer Authorized By:						
Attorney Docket Number:	AQI.002US1					
Receipt Date:	03-MAR-2008					
Filing Date:	10-DEC-2003					
Time Stamp:	18:03:52					
Application Type:	Utility under 35 USC 111(a)					

# Payment information:

Submitted wi	th Payment	yes	yes					
Payment Typ	e	Deposit Account	Deposit Account					
Payment was	successfully received in RAM	\$60	\$60					
RAM confirm	ation Number	3074	3074					
Deposit Acco	unt	160631	160631					
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	Claims	4		8	
	Applicant Arguments/Remarks	9		12	
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Information:					
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This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

## New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

### National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

## New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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(37 CFR 1.16(k), (i), or (m))			N/A				N/A				
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١D	EPENDENT CLAIM CFR 1.16(h))	S		m	nus 3 = *		X \$ =		1	X\$ =	
APPLICATION SIZE FEE     (37 CFR 1.16(s))     If the specification and drawings exceed 100     sheets of paper, the application size fee due     is \$250 (\$125 for small entity) for each     additional 50 sheets or fraction thereof. See     35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).     MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))				n size fee due for each n thereof. See							
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process) an application. Continentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.** If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2

**JA437** 

## PATENT APPLICATION

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of: Senkiw Attorney Docket No.: 4056.02US01 Confirmation No.: 7020 Application No.: 10/732,326 Filed: December 10, 2003 For: FLOW-THROUGH OXYGENATOR

## SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

Mail Stop Amendment Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Pursuant to 37 CFR § 1.56, and in addition to information disclosed in Applicant's Information Disclosure Statement filed July 19, 2004, the attention of the Patent and Trademark Office is hereby directed to the references listed on the attached Form PTO-1449. It is respectfully requested that the information be expressly considered during the prosecution of this application, and that the references be made of record therein and appear among the "References Cited" on any patent to issue therefrom. The listing of a reference herein is not an admission that the reference is prior art or that the reference is material to patentability.

This Information Disclosure Statement is being filed more than three months after the U.S. filing date and after the mailing date of a Final Action, Notice of Allowance or an action that otherwise closes prosecution in the application but before payment of the Issue Fee. Applicant hereby petitions that the Information Disclosure Statement be considered. Please

## Application No. 10/732,326

charge the \$180.00 petition fee under 37 CFR § 1.17(p) to Deposit Account No. 16-6031. Please credit or debit Deposit Account No. 16-0631 as needed to ensure consideration of the disclosed information.

I hereby certify that each item of information contained in this Information Disclosure Statement was cited in a communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of this Information Disclosure Statement. 37 CFR § 1.97(e)(1). A copy of the European Search Report is enclosed for the Examiner's convenience.

Respectfully submitted, J. Paul Haun Registration No. 53,003

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2

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 442 of 1320

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 443 of 1320

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# INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT) (51) International Patent Classification <sup>6</sup>: WO 99/39561 (11) International Publication Number: A01B 39/00 **A1** (43) International Publication Date: 12 August 1999 (12.08.99) (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). PCT/US99/02779 (21) International Application Number: (22) International Filing Date: 9 February 1999 (09.02.99) (30) Priority Data: 10 February 1998 (10.02.98) 09/021,721 US (71)(72) Applicant and Inventor: MAZZEI, Angelo, L. [US/US]; 11101 Mountain View Road, Bakersfield, CA 93307 (US). (74) Agents: PACIULAN, Richard, J. et al.; Ladas & Parry, Suite 2100, 5670 Wilshire Boulevard, Los Angeles, CA 90036-5679 (US). Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments. (54) Title: BENEFICIATION OF SOIL WITH DISSOLVED OXYGEN FOR GROWING CROPS REG 25 20 33 34 (57) Abstract An apparatus for beneficiation of soil by infusion of a treatment gas into a pressurized irrigation stream. The apparatus includes a cavitating venturi-type mixer-injector (35) with; a flow passage therethrough, an inlet and an outlet, a constricting portion of decreasing diameter, a cylindrical injector portion for injecting treatment gas into the flow passage, an increasing diameter expanding portion, an impermeable elongated conduit for receiving water and treatment gas from the mixer-injector (33) and a plurality of flow-restricting outlets disposed along the conduit wall permitting limited flow of water without substantial loss of pressure in the conduit. The mixer-injector is adapted to be connected to water source under pressure. Treatment gas will be infused into the water as it flows through the flow passage in the mixer-injector. The water will remain under super-atmospheric pressure until after it passes through the flow-restricting outlet.

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PCT/US99/02779

# BENEFICIATION OF SOIL WITH DISSOLVED OXYGEN FOR GROWING CROPS

#### Field of the Invention

This invention relates to the beneficiation of soil in which a crop is grown by supplying beneficial gases to the root region.

## Background of the Invention

Growing plants require water, oxygen and potentially other gases to support their life cycle. The water for most crops is derived from interstices in soil, which obtain it from rainfall or irrigation. Oxygen and other gases are obtained from the interstices, either from atmospheric gases which have migrated into the soil or from water in which the gases are dissolved. Although many gases can be beneficial to various crops, oxygen is one of the more important ones, and hence it will be dealt with specifically in this discussion, but this invention is not to be limited to the beneficial affects of oxygen only.

Generally, the concentration of oxygen or other gases in irrigation water is limited to that which is dissolved in accordance with Henry's Law. This is adequate for growth of crops. Farmers and growers of plants are fully aware of the range of wetness that a plant can tolerate. If the soil is maintained too wet for a substantial length of time, it will partially suffocate the microbial activity necessary for plant food conversion for plant uptake. It will, in effect drown. Too long a dry period will result in a lack of moisture to support plant transpiration even though there is plenty of oxygen in the soil.

Accordingly, with present practices, a plant grows best when it is alternately wetted and permitted to approach dryness. The grower attempts to provide adequate moisture and adequate oxygen to support varying transpiration ratios due to fluctuating weather conditions. Commercial intensive agricultural practice supplies oxygen as a function of the correct supply of oxygen dissolved in water, and as a function of air which is drawn into the soil as water is withdrawn from the interstitial spaces in the soil. However these sources can be quite variable. Well water, for example, tends to have less dissolved oxygen, and often contains undesirable other gases. The oxygen content in water supplied in ditches and furrows can vary depending on

1

#### PCT/US99/02779

water temperatures and ambient conditions.

It is an object of this invention to control and to improve the supply of oxygen to the soil, and thereby improve the growth of the plant or crop. The terms "plant" and "crop" are used interchangeably herein.

This is a fundamental problem, and efforts have been made and suggested to improve the oxygen supply by aerating irrigation water. This can indeed increase the oxygen concentration in the water, but it does not address the issue of what gas content actually reaches all plants in an area of significant size. Previous efforts have achieved some disappointingly limited improvement. For example they have not provided an optimal increase in production by weight, uniform improvement over a substantial area, or significant advancement of maturity of the plants. Their small improvement has been commercially insignificant.

This invention does provide these improvements. For example, adjacent plots of land near Bakersfield, California were planted with bell pepper plants spaced about 12-14 inches (30-36 cm) apart, along raised rows about 620 feet (200 m) long between furrows spaced about 40 inches (1 m) apart. A test plot was prepared according to this invention, as will be described below. The control plot was planted the same way, with an identical irrigation system but without the air supply of this invention. The systems were operated identically.

The results were surprisingly and unpredictably favorable. For example, it was found that the peppers reached a given point of maturity with this invention about one week sooner than peppers in the control plot. This was confirmed by observing the presence of a larger proportion of red peppers to green peppers in the test plot sooner than in the control plot. This is not a small matter. Especially at the start of a season, the earlier produce commands immediate purchase and at a premium price. This premium goes straight to the bottom line as profit. In addition an increase in production in weight of crops over the full season of about 5.6% over the control plot was noted, which also is a direct profit from this invention.

Because this invention's effects are substantially uniform over the entire field, maximum production from a plot of significant size can be anticipated. While bell peppers are given as an example of the results of using this invention, other crops may expect beneficial results, also.

As a further advantage, the plants are less stressed while growing and producing. The

2

#### PCT/US99/02779

average stress index of the control plot for the season was about -5.52, while the test plots had an index of about -5.76. A larger negative number is the better.

It is an object of this invention to provide a practical means to beneficiate the soil for the above purpose. By the term "beneficiate" is meant addition of a substance to improve the soil's microbial activity for better plant food uptake as a total body by the injection of air/oxygen into the vital root zone area of the plant including moisture for the intended purpose. It is not used in the sense of the addition of a chemical such as gypsum or fertilizer which is used to change the chemical constituents of the soil itself.

## Brief Description of the Invention

A system to beneficiate soil according to this invention is intended to supply water and air/oxygen along with other potentially beneficial gases to the subsurface root region of the plant. It is intended to be useful over a substantial area of cropland. As an example, an area of 4.8 acres (19,400 m<sup>2</sup> or 1.94 ha), with rows as long as 620 (200m) feet, and from a single supply, as many as 98 of these rows can readily be treated by this invention. Treated water is to be released beneath the surface of the soil or beneath a covering for the soil such as a mulch. A plastic sheet is regarded as a mulch, although its principal purpose is to control weeds.

An example of a system for this purpose is drip irrigation in which water under system pressure is released through spaced-apart emitters directly into the subsurface soil near the plant rather than being applied to the surface or in furrows. Until the water is released from the emitter, it remains under system pressure so that it contains more dissolved oxygen and other gases than it would under atmospheric pressure, and it will also contain very small micro bubbles of oxygen and other gases such as nitrogen which have not dissolved, especially when air is used as the source of oxygen. As a consequence, when this water is released from system pressure to atmospheric pressure, the released water will then carry a dissolved amount of oxygen respective to this lower pressure, and will release in the soil the excess oxygen which was dissolved at system pressure. It will also release such gases including oxygen as may have existed in micro bubbles.

Importantly, because the system is under pressure, the quality of the mixture of oxygenrich water and micro-bubbles remains substantially uniform throughout the entire pressured

3

#### PCT/US99/02779

system. Some coalescencing of the micro-bubbles can be expected, but because of their small size and dispersion, coalescence into major bubbles will not be appreciable. Therefore the water delivered through all exit orifices is substantially uniform so that every plant will be treated consistently.

It will be noted that this arrangement will compensate for the absence of oxygen from the atmosphere into the soil when covered by an impermeable plastic sheet (mulch).

According to one aspect of this invention, oxygen, oxygen containing gases such as air, and other gases beneficial for soil treatment, are injected into the water stream through a mixerinjector. The mixer-injector has a flow passage therethrough with a constricting portion, an injection portion, and an expanding portion in that order. Treatment gas enters the injection portion through an injection port.

The mixer-injector is a cavitating type which produces a reduced pressure in the injection portion, and turbulence in the injection portion. The turbulence disperses the treatment gas throughout the stream. It also reduces the size of the bubbles while it also increases their number.

According to a preferred but optional feature of this invention, the turbulence, distribution and reduction in bubble size may be improved by providing twisting vanes in the constricting portion and straightening vanes in the expanding portion. The stream from the expanding portion proceeds to a user system which may include one or more manifolds, and from there through tubing to points of discharge. It is maintained under pressure until it leaves the tubing. The rate of flow through this system and its length allow sufficient time for the oxygen or other gases to be dissolved to saturation level. The bubbles produced by this mixerinjector are small enough that they do not appreciably coalesce or rise to a surface. They tend to be discharged through the emitter along with the water.

According to a preferred but optional feature of the invention, the treatment gas is air, used for its oxygen content, as well as other potentially beneficial gases.

The above and other features of this invention will be fully understood from the following detailed description and the accompanying drawings, in which:

4

#### PCT/US99/02779

#### Brief Description of the Drawings

Fig. 1 is schematic illustration of an irrigation system according to the preferred embodiment of this invention;

Fig. 2 is a schematic cross-section of a localized region where water is being emitted;

Fig. 3 is an axial cross-section of the mixer-injector used in this invention;

Fig. 4 is a left hand end view of Fig. 3;

Fig. 5 is a right hand end view of Fig. 3; and

Fig. 6 is a longitudinal cross-section of a fragment of typical drip irrigation tubing showing an emitter suitable for use with this invention; and

Fig. 7 is a cross-section taken at line 7-7 in Fig. 6.

## Detailed Description of the Invention

The object of this invention is to provide a supply of water and oxygen and/or other potentially beneficial gases to soil in the root area of a growing plant. The presently-preferred example of its use is in drip irrigation where, as shown in Fig. 2, a plant 20 is grown in soil 21 having a surface 22. For some crops, a plastic sheet 23 (regarded as a "mulch" for its weed resisting capability) or organic mulch is applied over the surface of the soil near the plant. Water supplied by this system will ordinarily be discharged about 6-10 inches (15-25 cm) below the soil surface.

As shown, a drip irrigation tubing 25 extends along a row 26. At intervals its wall is pierced by emitters 27 (Fig. 6). An emitter is simply an orifice of some kind through which water will flow from the tubing into the soil region at a regulated rate, under designated system pressure. There is a pressure drop across the emitters from system pressure, usually about 10-20 psig to atmospheric pressure.

The pressure drop at the discharge sites is of considerable importance to this invention. The reason is the higher concentration of oxygen and/or other gases that exist in the water at the higher pressure according to Henry's law. However, there is more to it than that, because frequently agricultural water supplies are not fully saturated with oxygen. In this invention, super saturation at atmospheric and system pressure can be assured, so that extra oxygen will be released from solution as the pressure drops, plus additional oxygen in the gas content of the micro bubbles that are produced. This water stream is very rich in the treatment gas.

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PCT/US99/02779

A water source 30, such as a well, a pond, or a water main supplies water to be pressurized by a pump 32 to system pressure. If the source is a main and pressure in the main is adequate, a pump will be unnecessary. Its output is provided to a distribution system 33 which includes such conventional valves, regulators, and other controls as may be suitable. The system may include headers 34 extending along the heads of the rows 26 of crops. From the headers, flexible drip irrigation tubings 25 extend along the rows or to any desired location where the emitters 27 are to be placed. Applications other than in rows is contemplated, for example in groups of emitters around a tree, and a series of trees.

The mixer-injector 35, best shown in Fig. 3 optimally produces the desired beneficiated water. It receives water from the pump and passes it to the distribution system. Injector-mixers such as those shown in Mazzei United States patent No.4,123,800, issued October 31, 1978 are cavitating types which will in fact increase the oxygen and/or other gases content of water by drawing gas into the stream, and creating micro-bubbles. Such mixer-injectors are useful in this invention. However, the increased turbulence and shear in the illustrated mixer-injector produces smaller micro-bubbles and distributes them better, thereby providing an improved and more stable mix. Both types of mixer-injectors assure that oxygen and/or other gas saturation can be attained, and that the bubbles will be so small that they will minimally gas-out of the water in the time span generally involved in flow through systems such as these, but the mixer-injector shown in Fig. 3 provides surprisingly-improved results.

Full details of mixer-injector 35 will be found in applicant's United States Patent No. 5,863,128 issued January 26, 1999 entitled "Mixer-Injectors With Twisting and Straightening Vanes" which is incorporated herein in its entirety for its detailed showing of the construction and theory of operation of this mixer-injector. For purposes of this invention, it is sufficient to describe its basic elements.

Full details of a less-effective, but still useful mixer-injector for use with this invention will be found in the said Mazzei patent No. 4,123,800, which is incorporated herein in its entirety for its showing of such a mixer-injector. It lacks certain vanes yet to be described, which provide important advantages.

Mixer-injector 35 has a body 36 with a flow passage 37 extending from an entry port 38

6

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### PCT/US99/02779

to an exit port 39. An internal wall 40 forming the flow passage includes, from the entry port in this order, a cylindrical entry portion 41, a constricting portion 42, an injection portion 43, and an expanding portion 44 which terminates at exit port 39.

An injection port 45 enter the injection portion near to the constricting portion. It preferably exists as a circumferential groove 46 in the internal wall, communicating with a passage 47 that receives treatment gas to be provided to the stream in the flow passage, for example from atmospheric air. A metering valve and a check valve 80 are placed in passage 45 to provide a unidirectional flow of the correct amount of oxygen.

It is convenient to regulate the pressure and flow rate by means of establishing a flow and pressure drop through a regulator valve (which may be a flow restrictors instead) in a bypass passage across the mixer-injector.

To this point, the mixer-injector shown in the said Mazzei '800 patent is described, and is useful. However, additional features as shown in the said Mazzei patent application provide importantly improved performance. These features are twisting vanes 48 in the constricting portion, and straightening vanes 49 in the expanding portion.

The twisting vanes 48 are provided as a group (eight is a useful number) of individual vanes with crests which as they extend along the central axis 50 of the flow passage also extend at an acute angle 51 to a plane passed through them and which includes the central axis. They rise from the entry portion into the constricting portion. They do not intersect the central axis. They give a twist to the outer region of the stream, so that when it crosses the injection port it has an increased turbulence caused by the confluence of the central "core" of the stream (which is not twisted) and the outer portion (which is twisted). This increased turbulence results in a more thorough mixing of the water and the treatment gas, and the reduction of size of the microbubbles, all to the advantage of this process.

Once this is attained, it is advantageous for the turbulence to be reduced, while still further shearing the micro-bubbles. This is accomplished by the group of straightening vanes 49, which extend along the expanding portion. They have crests 51 that are preferably parallel to the central axis, and are spaced apart from it. From exit port 39, the stream enters the distribution system extending to the plants.

7

#### PCT/US99/02779

As previously stated, emitters 27 are placed along the length of the tubing. Their characteristic is to provide for a slow bleed of water from the tubing into the subsoil. Such emitters are well-known and of several types. They constitute, in effect, a bleed valve that permits a very limited rate of flow of fluid therethrough, so the pressure in the tubing is not materially reduced, and the contents remain under pressure throughout the system.

A typical useful emitter 60 is shown formed as part of a drip irrigating tubing 61. In this emitter, the main passage 62 is formed by a wall 63. Longitudinal edges 64, 65 of the wall are overlapped to leave a restricted channel 66 between overlapped margins 67, 68. A series of inlet ports 69 is formed from passage 62 into channel 66. These ports are small and enter at numerous locations along the overlaps. The channel may further restricted by internal diverters or by serpentine passages which further reduce the rate of flow of water through channel 66. At one end of channel 66, a longitudinal slit 71 through the outer overlap releases the water from channel 66 to atmosphere.

There are other types of emitters, including small orifices through the wall of the tubing. Any emitter capable of establishing a regulated rate of fluid flow from passage 62 is acceptable.

The operation of this system will be evident from the foregoing. With the desired throughput of water per unit time decided upon, a suitably sized mixer-injector will be selected and plumbed into the system. The flow through the injection portion will establish a subatmospheric pressure in that portion which will draw treatment gas into the injection portion. The rate of flow of this gas will be adjusted by valve 55 to pass the gas at a suitable flow rate for the purposes intended.

Some treatment gas drawn into the mixer-injector will be dissolved and the remainder will be divided into micro-bubbles as described, and will flow into the system, ultimately to and through the emitters. The drip irrigation tubing is impermeable. Water and gases can leave only through the emitters (or other flow-limiting outlets, of which emitters and orifices are only two examples). The existing fluids have substantially the same water/gas mix at that point as at all other locations in the system downstream from the mixer-injector.

In a system as previously described, water was supplied to a two 2 inch mixer-injector sold by Mazzei Injector Corporation as its part No. 2081. It is constructed a shown in Fig. 3,

## 8

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### PCT/US99/02779

and as further disclosed in detail in the said Mazzei patent application. Water flowed through the system at the rate of about 260 gallons per minute and air was drawn into it at the rate of approximate 3.5 SCFM. Flow was intermittently supplied, on the average about 2 hours every 3 days and more frequently as the plant grows.

Accordingly, the soil will be beneficiated by the concurrent addition of water and treatment gas. It provides an improvement in growing conditions because it ultimately promotes a healthier plant root. Systems can be provided with the use of this invention which allow far less plant stress under varying weather conditions and watering intervals.

This invention is not to be limited by the embodiments shown in the drawings and described in the description, which are given by way of example and not of limitation, but only in accordance with the scope of the appended claims.

9

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 454 of 1320

WO 99/39561

#### PCT/US99/02779

I CLAIM:

1. Apparatus for beneficiation of soil by infusion of a treatment gas into a pressurized irrigation stream, said apparatus comprising:

a cavitating venturi-type mixer-injector having a body with an internal wall forming a flow passage therethrough, said flow passage having a central axis, an inlet, an outlet, and between said inlet and outlet said wall forming a constricting portion of decreasing diameter, a substantially cylindrical injector portion, and an expanding portion having an increasing diameter all as they progress in that order from inlet to outlet, an injection port, said injector port receiving treatment gas from a source of gas for injection into said flow passage; an impermeable elongated conduit having a length and a peripheral wall

forming a longitudinal passage for receiving water and treatment gas from said mixer-injector; a plurality of flow-restricting outlets disposed along the length of said

conduit wall, passing through said conduit wall to permit limited flow of water from said longitudinal passage without substantial loss of pressure in said conduit;

said mixer-injector adapted to be connected to a source of water under pressure, whereby treatment gas will be infused into said water as it flows through the flow passage in the mixer injector, and said water will remain under super-atmospheric pressure until after it passes through a said flow-restricting outlet.

2. Apparatus according to claim 1 in which said flow restricting outlets are emitters either internal or external.

3. Apparatus according to claim 1 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

4. Apparatus according to any one of claims 1-3 in which a plurality of straightening vanes extend along at least a part of the expanding portion, said straightening vanes being

10

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### PCT/US99/02779

parallel to said central axis, but being spaced from said central axis.

5. Apparatus according to claim 4 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

6. Apparatus according to claim 5 in which said flow restricting outlets are emitters, either internal or external.

7. In combination:

crop growing soil in which a rooted crop is to be grown, said soil having a top surface; and

apparatus for beneficiation of said soil by infusion of a treatment gas into a pressurized irrigation stream of water, said apparatus comprising:

a cavitating venturi-type mixer-injector having a body with an internal wall forming a flow passage therethrough, said flow passage having a central axis, an inlet, an outlet, and between said inlet and outlet said wall forming a constricting portion of decreasing diameter, a substantially cylindrical injector portion, and an expanding portion having an increasing diameter all as they progress in that order from inlet to outlet, an injection port, said injector port receiving treatment gas from a source of gas for injection into said flow passage; an impermeable elongated conduit having a length and a peripheral wall

forming a longitudinal passage for receiving water and treatment gas from said mixer-injector; a plurality of flow-restricting outlets disposed along the length of said

conduit wall beneath said top surface, passing through said conduit wall to permit limited flow of water from said longitudinal passage without substantial loss of pressure in said conduit;

said mixer-injector adapted to be connected to a source of water under pressure, whereby treatment gas will be infused into said water as it flows through the flow passage in the mixer injector, and said water will remain under super atmospheric pressure until

11

#### PCT/US99/02779

after it passes through a said flow-restricting outlet.

8. Apparatus according to claim 7 in which said flow restricting outlets are emitters, either internal or external.

9. Apparatus according to claim 7 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

10. Apparatus according to any one of claims 7-9 in which a plurality of straightening vanes extend along at least a part of the expanding portion, said straightening vanes being parallel to said central axis, but being spaced from said central axis.

11. Apparatus according to claim 10 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

12. Apparatus according to claim 11 in which said flow restricting outlets are emitters either internal or external.

13. A method to improve growing conditions for crops which are grown in soil that has a top surface:

utilizing apparatus for beneficiating soil by infusion of a treatment gas into an irrigation stream of water, said apparatus comprising:

a cavitating venturi-type mixer-injector having a body with an internal wall forming a flow passage therethrough, said flow passage having a central axis, an inlet, an outlet,

12

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### PCT/US99/02779

and between said inlet and outlet said wall forming a constricting portion of decreasing diameter, a substantially cylindrical injector portion, and an expanding portion having an increasing diameter all as they progress in that order from inlet to outlet, an injection port, said injector port receiving treatment gas from a source of gas for injection into said flow passage;

an impermeable elongated conduit having a length and a peripheral wall forming a longitudinal passage for receiving water and treatment gas from said mixer-injector;

a plurality of flow-restricting outlets disposed along the length of said conduit wall, passing through said conduit wall to permit limited flow of water from said longitudinal passage without substantial loss of pressure in said conduit;

said mixer-injector adapted to be connected to a source of water under pressure, whereby treatment gas will be infused into said water as it flows through the flow passage in the mixer injector, and said water will remain under super atmospheric pressure until after it passes through an emitter buried in said conduit beneath said top surface;

forcing a stream of water under pressure into the entry port of said mixer injector, while admitting air into said injection portion through said injection port, thereby providing in said longitudinal passage a water stream under atmospheric pressure enriched with treatment gas, said emitters permitting limited flow of said stream into said soil.

14. The method according to claim 13 in which said flow restricting outlets are emitters.

15. The method according to claim 13 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

16. The method according to any one of claims 13-15 in which a plurality of straightening vanes extend along at least a part of the expanding portion, said straightening vanes being parallel to said central axis, but being spaced from said central axis.

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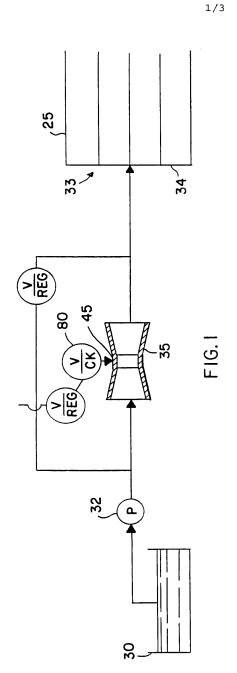
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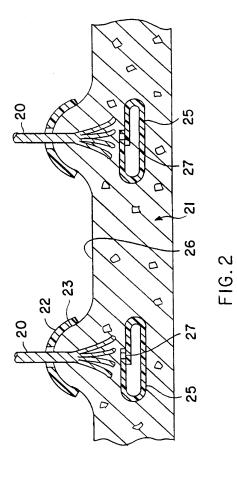
17. The method according to claim 16 in which a plurality of twisting vanes extend along at least a part of said constricting portion, said vanes extending axially but at an acute angle relative to an imaginary plane which passes through them and which includes the central axis, said twisting vanes extending toward said central axis, but being spaced from said central axis.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

PCT/US99/02779

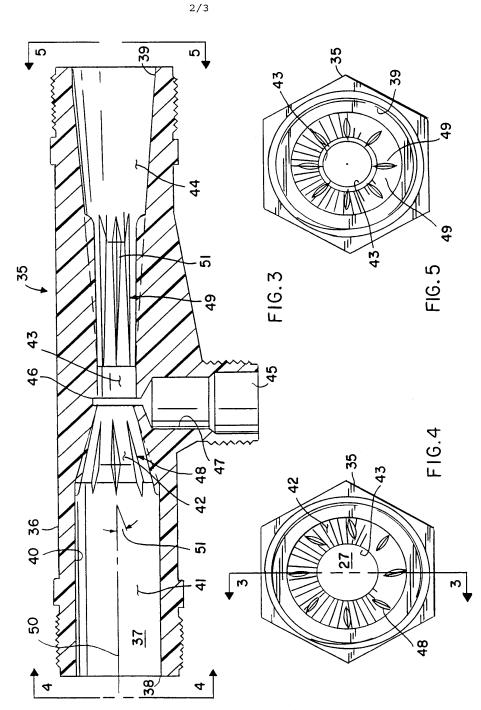




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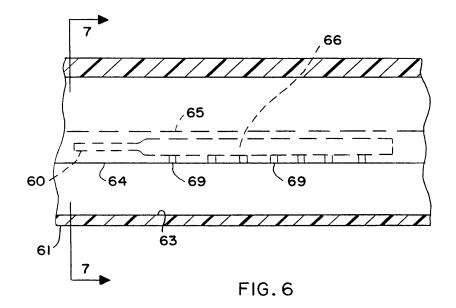


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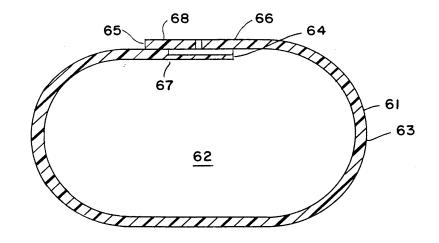


FIG.7

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 462 of 1320

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/02779

CLASSIFICATION OF SUBJECT MATTER А. IPC(6) :A01B 39/00 US CL :47/58,1.01F

According to International Patent Classification (IPC) or to both national classification and IPC В. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 47/58, 48.5, 1.01F; 366/150, 163.2

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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Category*	Citation of document, with indication, where a	opropriate, of the relevant passages	Relevant to claim No	
A	US 1,200,869 A (RIFE) 10 October 1 pages 1-2 and Figures 1-2.	916 (10.10.16),	1,7,13 2,6,8,12,14,17	
X,P	US 5,863,128 A (MAZZEI) 26 January 1999 (26.01.99), 3-5, cover page Abstract and Figure 1.			
Y	US 5,697,187 A (PERSINGER) 16 D cover page Abstract and Figure-1.	1,7,13		
Y	US 3,046,747 A (TIMPE) 31 July 19 see entire document, especially colum		1,7,13 2,6,8,12,14,17	
X Furth	er documents are listed in the continuation of Box (	C. See patent family annex.		
	ecial categories of cited documents:	"T" later document published after the in	ternational filing data or priority	
A* do	cument defining the general state of the art which is not considered be of particular relevance	date and not in conflict with the ap the principle or theory underlying the	pplication but cited to understan the invention the claimed invention cannot b idered to involve an inventive ste the claimed invention cannot b	
E* ea	lier document published on or after the international filing date	"X" document of particular relevance; t considered novel or cannot be consid		
cit	cument which may throw doubts on priority claim(s) or which is ed to establish the publication date of another citation or other scial reason (as specified)	"Y" document of particular relevance; t		
sp		considered to involve an inventiv	e step when the document is ch documents, such combination	
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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 463 of 1320

	INTERNATIONAL SEARCH REPORT	International appl PCT/US99/0277	
C (Continua	tion). DOCUMENTS CONSIDERED TO BE RELEVANT	1	
Category*	Citation of document, with indication, where appropriate, of the relev	ant passages	Relevant to claim No
Y	US 4,123,800 A (MAZZEI) 31 October 1978 (31.10.7) cover page Abstract, columns 3-4 and Figure-1.	3),	1,7,13

Form PCT/ISA/210 (continuation of second sheet)(July 1992)\*

#### (12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: MICROBUBBLES OF OXYGEN

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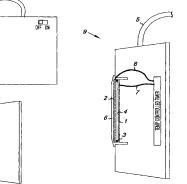
(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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VC, VN, YU, ZA, ZM, ZW.

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



(57) Abstract: An oxygen emitter (6) which is an electrolytic cell is disclosed. When the anode (1) and cathode (2) are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are generated. The hydrogen forms bubbles at the cathode, which bubbles rise to the surface. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium. Models suitable for different uses are disclosed.

#### WO 03/072507

#### PCT/US03/05288

## MICROBUBBLES OF OXYGEN

#### FIELD OF THE INVENTION

5 This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of aqueous media.

### **BACKGROUND OF THE INVENTION**

- 10 Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and
- 15 hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium.
- 20 Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora. Contaminated water is
- 25 described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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#### WO 03/072507

#### PCT/US03/05288

atmosphere. Attempts have been made to reduce the size of the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. United States Patent Number 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3

millimeters in diameter. United States Patent Number 6,394,429 discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at high pressure through a small orifice.

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When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4 e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$
NET REACTION:	$6H_2O \rightarrow 4OH^- + 4H^+ + 2H_2 + O_2$

286 kilojoules of energy is required to generate one mole of oxygen.

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The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

2

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 467 of 1320

#### WO 03/072507

#### PCT/US03/05288

Many cathodes and anodes are commercially available. United States Patent
Number 5,982,609 discloses cathodes comprising a metal or metallic oxide of at
least one metal selected from the group consisting of ruthenium, iridium, nickel,
iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead,
titanium, platinum, palladium and osmium. Anodes are formed from the same
metallic oxides or metals as cathodes. Electrodes may also be formed from alloys
of the above metals or metals and oxides co-deposited on a substrate. The cathode
and anodes may be formed on any convenient support in any desired shape or size.
It is possible to use the same materials or different materials for both electrodes.
The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive
electrolytic water. An especially preferred anode disclosed in U. S. Patent Number

4,252,856 comprises vacuum deposited iridium oxide.

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Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and

20 additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

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### **RELATED APPLICATIONS**

This application claims priority of United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

WO 03/072507

PCT/US03/05288

## SUMMARY OF THE INVENTION

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

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Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

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### PCT/US03/05288

# **DESCRIPTION OF THE DRAWINGS**

Figure 1 is the  $O_2$  emitter of the invention.

5 Figure 2 is an assembled device.

Figure 3 is a diagram of the electronic controls of the O<sub>2</sub> emitter.

Figure 4 shows a funnel or pyramid variation of the O<sub>2</sub> emitter.

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Figure 5 shows a multilayer sandwich  $O_2$  emitter.

## **DETAILED DESCRIPTION OF THE INVENTION**

15 Definitions:

For the purpose of describing the present invention, the following terms have these meanings:

"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

" $O_2$  emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

25 "Microbubbles" means a bubble with a diameter less than 50 microns.

"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

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PCT/US03/05288

"Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

- <sup>5</sup> "Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.
- 10 The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen, O<sub>2</sub>. In the special dimensions of the invention, as explained in more detail in the following examples, O<sub>2</sub> forms bubbles which are too small to break the surface tension of the fluid. These bubbles remain suspended
- 15 indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the H<sub>2</sub> formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

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The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 471 of 1320

WO 03/072507

PCT/US03/05288

the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

# **Example 1.** Oxygen emitter:

- 5 As shown in Figure 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, OH). The cathode 2 is a 1/16 inch mesh marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage of gas and mixing of anodic and cathodic water
- and connected to a power source through a connection point 5. Figure 2 shows a plan view of the assembled device. The O<sub>2</sub> emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin
- 15 enough to separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is 0.050 inches thick. The spacer may be any
- 20 nonconductive material such as nylon, fiberglass, Teflon® polymer or other plastic. Because of the criticality of the space distance, it is preferable to have a noncompressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

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In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.05 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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PCT/US03/05288

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most economically. Figure 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

### Example 2. Measurement of O<sub>2</sub> bubbles.

Attempts were made to measure the diameter of the O<sub>2</sub> bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high

- contrast, backlit photograph of treated water with a millimeter scale reference was
   shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.
- 25 Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be hydrogen bubbles. When multiplied by the scale multiplier, the assumed O<sub>2</sub> bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the

8

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 473 of 1320

### WO 03/072507

#### PCT/US03/05288

nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the sensitivity of measurement so that sub-micron diameter bubbles can be measured.

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## Example 3. Other models of oxygen emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the

15 emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. Figure 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large 20

volumes of water rapidly.

The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at

9

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 474 of 1320

WO 03/072507

### PCT/US03/05288

higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of models suitable to various uses.

Watts
0.36
1.44
1.44
2.16
2.16
3.48
4.92
28.80
30.00
-

TABLE I

10

15

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# Example 4. Multilayer sandwich O<sub>2</sub> emitter

An O<sub>2</sub> emitter was made in a multilayer sandwich embodiment. (Figure 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 475 of 1320

### WO 03/072507

#### PCT/US03/05288

held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5 with a nylon washer 6. The dimensions selected were:

	•	cathode screen	0.045 inches thick		
5	•	nylon spacer	0.053 inches thick		
	•	anode grid	0.035 inches thick		
	•	nylon spacer	0.053 inches thick		
	•	cathode screen	0.045 inches thick,		
	for an overall emitter thickness of 0.231 inches.				

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If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

11

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 476 of 1320

WO 03/072507

PCT/US03/05288

I claim:

5 Claim 1. An emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other.

Claim 2. The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support.

15 Claim 4. The emitter of claim 1 wherein the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 5. The critical distance of claim 1 which is 0.005 to 0.140 inches.

20 Claim 6. The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 7. An emitter for electrolytic generation of microbubbles of oxygen comprising a plurality of anodes separated at a critical distance from a
plurality of cathodes and a power source all in electrical communication with each other.

Claim 8. A method for keeping aquatic animals emitter alive comprising inserting the emitter of claim 1 or claim 7 into the aquatic medium of the aquatic animals.

12

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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15

#### PCT/US03/05288

Claim 9. The method of claim 8 wherein the aquatic animal is a fish.

Claim 10. The method of claim 8 wherein the aquatic animal is a shrimp.

Claim 11. A method for lowering the biologic oxygen demand of polluted water comprising passing the polluted water through a vessel containing the emitter of claim 1.

10 Claim 12. The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

Claim 13. An emitter for electrolytic generation of microbubbles of oxygen comprising a platinum-iridium oxide anode on a titanium support separated at a critical distance of from 0.045 inches to 0.060 inches from a stainless steel

screen 1/16 inch thick cathode all in electrical communication with a battery.

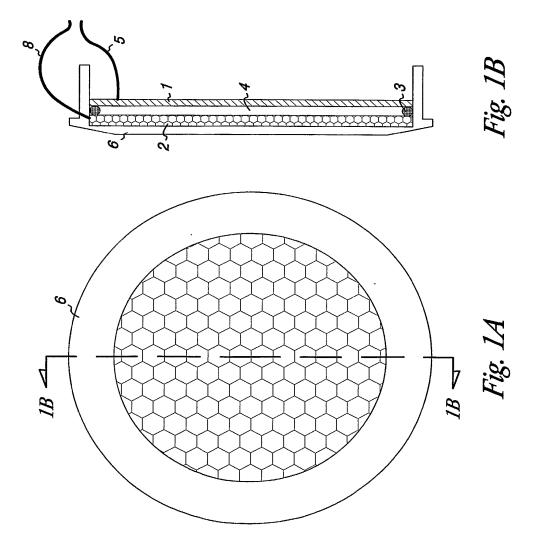
Claim 14. The emitter of claims 1, 7 or 13 further comprising a timer control.

13

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

PCT/US03/05288

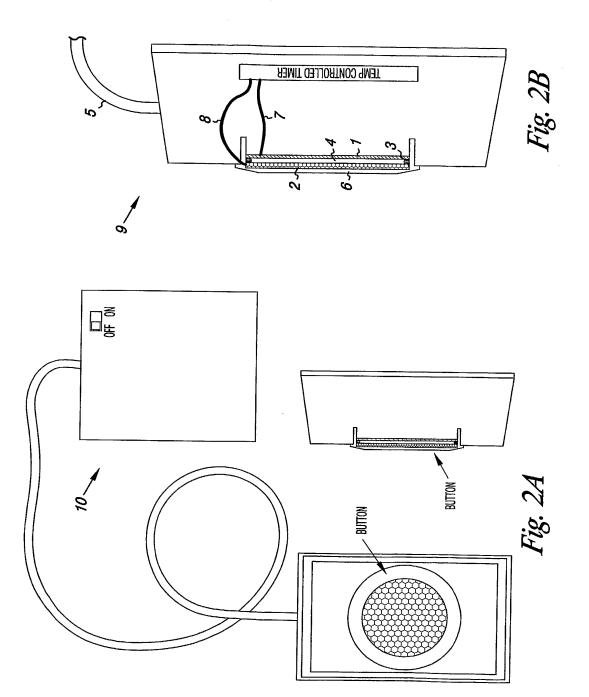




SUBSTITUTE SHEET (RULE 26)

JA476

PCT/US03/05288



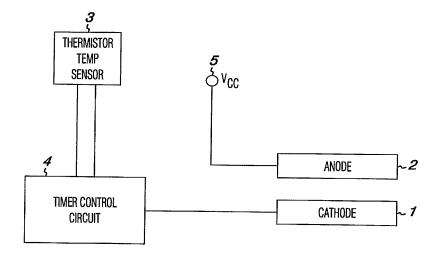
2/5

SUBSTITUTE SHEET (RULE 26)

JA477

PCT/US03/05288

3/5

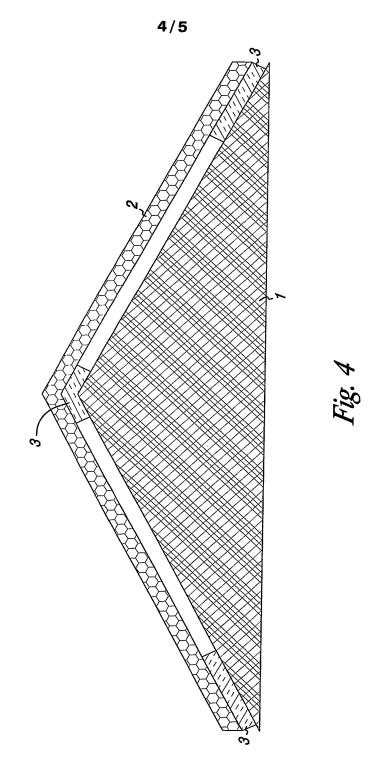


*Fig. 3* 

# SUBSTITUTE SHEET (RULE 26)

JA478

PCT/US03/05288



# SUBSTITUTE SHEET (RULE 26)

JA479

PCT/US03/05288



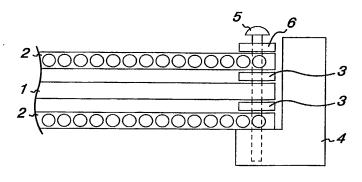


Fig. 5A

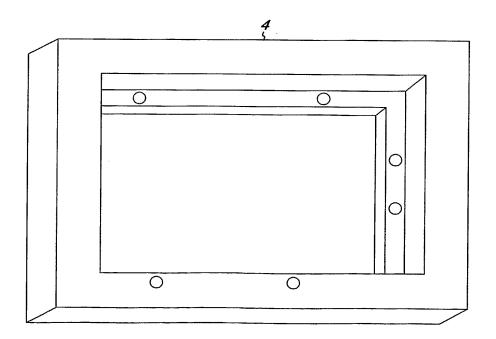


Fig. 5B

# SUBSTITUTE SHEET (RULE 26)

JA480

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 483 of 1320

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	INTERNATIONAL SEARCH REPOR	Т	International app			
			PCT/US03/05	288		
A. CLA	SSIFICATION OF SUBJECT MATTER					
IPC(7)	:C02F 1/30					
	:210/748, 758; 204/554, 232, 660					
	to International Patent Classification (IPC) or to both	national classificatio	n and IPC			
	LDS SEARCHED	hu dogification	hola			
	locumentation searched (classification system followed	by classification syn	10015)			
U.S. :	210/748, 758; 204/554, 232, 660					
Documenta	tion searched other than minimum documentation to	the extent that suc	h documents are i	included in the fields		
searched		,				
Electronic o	data base consulted during the international search (n	ame of data base and	, where practicable	e, search terms used)		
WEST						
		(				
C. DOC	UMENTS CONSIDERED TO BE RELEVANT	·	······································			
<u>c.</u> <u>b</u> cc						
Category*	Citation of document, with indication, where ap	propriate, of the relev	ant passages	Relevant to claim No.		
Х	US 5,993,618 A (SCHULZE et al) 30 I	November 1999,	Fig. 11, col.	1-4,7		
	5, line 49-col. 6, line 7, abstract, col.		U I			
А				5,6,8-14		
X,P	US 6,368,592 B1 (COLTON et al) 09	April 2002 col	7-8	1,2,4		
		April 2002, col	. 7-0.	1,2,4		
A,P	3,5-14					
, -						
Furt	l her documents are listed in the continuation of Box (	C. See pater	nt family annex.			
"A" do	<ul> <li>Special categories of cited documents: "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</li> </ul>					
	nsidered to be of particular relevance			e claimed invention cannot be		
	rlier document published on or after the international filing date cument which may throw doubts on priority claim(s) or which is		vel or cannot be conside ument is taken alone	red to involve an inventive step		
cit	ed to establish the publication date of another citation or other ecial reason (as specified)			e claimed invention cannot be		
"O" da	cument referring to an oral disclosure, use, exhibition or other	combined with	1 one or more other suc	step when the document is h documents, such combination		
	eans	-	to a person skilled in			
than the priority date claimed						
Date of the	actual completion of the international search	Date of mailing of t		1		
02 JUNE 2003 <b>11</b> JUN 2003						
Name and	mailing address of the ISA/US	Authorized officer	<u> </u>	r lon.		
Box PCT	oner of Patents and Trademarks	BETSEV M U	FY / Minh	Nally		
Washingto Facsimile N	on, D.C. 20231 No. (703) 305-3930					
r acomme r	No. (703) 305-3230	Telephone No. (	100/ 000-0001	/		

Form PCT/ISA/210 (second sheet) (July 1998)\*

Electronic Patent Application Fee Transmittal					
Application Number:	10732326				
Filing Date:	10	-Dec-2003			
Title of Invention:	Flow-through oxygenator				
First Named Inventor/Applicant Name:	Ja	mes Andrew Senk	iw		
Filer:	J.	Paul Haun/Valerie	Mitchell		
Attorney Docket Number:	AC	QI.002US1			
Filed as Large Entity					
Utility Filing Fees					
Description		Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:					
Pages:					
Claims:					
Miscellaneous-Filing:					
Petition:					
Patent-Appeals-and-Interference:					
Post-Allowance-and-Post-Issuance:					
Extension-of-Time:					

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 485 of 1320

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Miscellaneous:				
Submission- Information Disclosure Stmt	1806	1	180	180
	Tota	al in USE	D (\$)	180

Electronic Acknowledgement Receipt					
EFS ID:	2873658				
Application Number:	10732326				
International Application Number:					
Confirmation Number:	7020				
Title of Invention:	Flow-through oxygenator				
First Named Inventor/Applicant Name:	James Andrew Senkiw				
Customer Number:	24113				
Filer:	J. Paul Haun/Valerie Mitchell				
Filer Authorized By:	J. Paul Haun				
Attorney Docket Number:	AQI.002US1				
Receipt Date:	18-FEB-2008				
Filing Date:	10-DEC-2003				
Time Stamp:	16:06:01				
Application Type:	Utility under 35 USC 111(a)				

# Payment information:

Submitted with Payment	yes				
Payment Type	Deposit Account				
Payment was successfully received in RAM	\$180				
RAM confirmation Number	7413				
Deposit Account	160631				
Authorized User					
The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:					
Charge any Additional Fees required under 37 C.F.R. Section 1.16 (National application filing, search, and examination fees)					
Charge any Additional Fees required under 37 C.F.R. Section 1.17 (Patent application and reexamination processing fees)					

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 487 of 1320

Charge	any Additional Fees required under 37	C.F.R. Section 1.21 (Miscellane	eous fees and charges)				
File Listin	g:						
Document Number	Document Description	File Name	File Size(Bytes) /Message Digest	Multi Part /.zip	Pages (if appl.		
1	Information Disclosure Statement	4056_02US01_IDS.pdf	654682			no	3
	(IDS) Filed		d9c9b81d28e51d091b3c232ca22e60e 5d0c68b26	110			
Warnings:							
Information:							
This is not an U	JSPTO supplied IDS fillable form						
	Foreign Deference	4056_02US01_EPSEARCH.	755715	20	2		
2	Foreign Reference	pdf	35d60ba736afce768adfdacfce0aafff47 062df0	no	3		
Warnings:							
Information:							
3	Foreign Reference	4056_02_WO09939561A1.p df	861803	no	21		
5			1e50a0449e0b0c90511a8d6a69e915431 1715a86		21		
Warnings:							
Information:							
4	Foreign Reference	4056_02_WO03072507A1.p	770577	no	20		
		df	b688350ba6f340216d46e4f90f431842f 9cc8dcb	110	20		
Warnings:							
Information:		1					
5	Fee Worksheet (PTO-06)	fee-info.pdf	8158	no	2		
			25337a4571114dbf510e675b4323bf157 ae605a0		_		
Warnings:							
Information:							
		Total Files Size (in bytes)	30	50935			

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

# New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

## National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

# New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

	<u>ed States Patent a</u>	AND TRADEMARK OFFICE	UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F P.O. Box 1450 Alexandria, Virginia 223 www.uspto.gov	Trademark Office OR PATENTS
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003	James Andrew Senkiw	AQ1.002US1	7020
	7590 11/01/2007	HRISTENSEN PA	EXAM	INER
4800 IDS CEN				
80 SOUTH 8TH MINNEAPOLI	H STREET S, MN 55402-2100		ART UNIT	PAPER NUMBER
	-,		1793	
		•	MAIL DATE	DELIVERY MODE
			11/01/2007	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

PTOL-90A (Rev. 04/07)

JA487

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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N N					
· · · · · · · · · · · · · · · · · · ·	Application No.	Applicant(s)			
	10/732,326	SENKIW, JAMES ANDREW			
Office Action Summary	Examiner	Art Unit			
	Lois Zheng	1793			
The MAILING DATE of this communication Period for Reply	n appears on the cover sheet v	vith the correspondence address			
A SHORTENED STATUTORY PERIOD FOR R WHICHEVER IS LONGER, FROM THE MAILIN - Extensions of time may be available under the provisions of 37 C after SIX (6) MONTHS from the mailing date of this communicatio - If NO period for reply is specified above, the maximum statutory p - Failure to reply within the set or extended period for reply will, by Any reply received by the Office later than three months after the earned patent term adjustment. See 37 CFR 1.704(b).	G DATE OF THIS COMMUN FR 1.136(a). In no event, however, may a n. veriod will apply and will expire SIX (6) MC statute, cause the application to become A	ICATION. reply be timely filed NTHS from the mailing date of this communication. BANDONED (35 U.S.C. § 133).			
Status					
1) Responsive to communication(s) filed on	17 August 2007.				
2a)⊠ This action is <b>FINAL</b> . 2b)□	This action is non-final.				
3) Since this application is in condition for all	owance except for formal ma	tters, prosecution as to the merits is			
closed in accordance with the practice un	der <i>Ex parte Quayle</i> , 1935 C.	D. 11, 453 O.G. 213.			
Disposition of Claims					
4) Claim(s) <u>1-26</u> is/are pending in the application	ation.				
4a) Of the above claim(s) <u>5-8 and 10-12</u> is	are withdrawn from consider	ation.			
5) Claim(s) is/are allowed.		· .			
6)⊠ Claim(s) <u>1-4,9,13 and 15-26</u> is/are rejecte	d.				
7) Claim(s) <u>14</u> is/are objected to.					
8) Claim(s) are subject to restriction a	ind/or election requirement.				
Application Papers					
9) The specification is objected to by the Exa	miner.				
10) The drawing(s) filed on is/are: a)	] accepted or b)  objected to	by the Examiner.			
Applicant may not request that any objection to	o the drawing(s) be held in abeya	ance. See 37 CFR 1.85(a).			
Replacement drawing sheet(s) including the c	orrection is required if the drawin	g(s) is objected to. See 37 CFR 1.121(d).			
11) The oath or declaration is objected to by the	ne Examiner. Note the attache	ed Office Action or form PTO-152.			
Priority under 35 U.S.C. § 119					
12) Acknowledgment is made of a claim for fo	reign priority under 35 U.S.C.	§ 119(a)-(d) or (f).			
a) All b) Some * c) None of:					
1. Certified copies of the priority docu					
2. Certified copies of the priority docu					
3. Copies of the certified copies of the priority documents have been received in this National Stage					
application from the International Bureau (PCT Rule 17.2(a)).					
* See the attached detailed Office action for	a list of the certified copies no	t received.			
Attachment(s)					
1) X Notice of References Cited (PTO-892)	4) 🗌 Interview	Summary (PTO-413)			
2) 🔲 Notice of Draftsperson's Patent Drawing Review (PTO-94	8) Paper No	o(s)/Mail Date			
3) Information Disclosure Statement(s) (PTO/SB/08) Paper No(s)/Mail Date	5) 🛄 Notice of 6) 🗌 Other:	Informal Patent Application			
U.S. Patent and Trademark Office	·				
PTOL-326 (Rev. 08-06) Off	ice Action Summary	Part of Paper No./Mail Date 20071029			

**JA488** 

Page 2

# DETAILED ACTION

# Status of Claims

1. Claims 1-4 and 9 are amended in view of applicant's amendment filed 17 August 2007. New claims 13-26 are added in view of applicant's amendment. Claims 5-8 and 10-12 remain withdrawn from consideration. Therefore, claims 1-4, 9 and 13-26 are currently under examination.

# Status of Previous Rejections/Objections

2. The rejection of claims 1-3 under 35 U.S.C. 102(e) as being anticipated by Zappi et al. US 6,328,875 B1(Zappi) is withdrawn in view of applicant's claim amendment filed 17 August 2007.

The rejection of claim 4 under 35 U.S.C. 103(a) as being unpatentable over Zappi is withdrawn in view of applicant's claim amendment filed 17 August 2007.

The rejection of claim 9 under 35 U.S.C. 103(a) as being unpatentable over Zappi in view of Cairns et al. US 4,587,001(Cairns) is withdrawn in view of applicant's claim amendment filed 17 August 2007.

3. The rejection of claims 1-4 under 35 U.S.C. 103(a) as being unpatentable over Divisek et al. US 4,225,401(Divisek) is withdrawn in view of applicant's claim amendment filed 17 August 2007.

The rejection of claim 9 under 35 U.S.C. 103(a) as being unpatentable over Divisek in view of Cairns et al. US 4,587,001(Cairns) is withdrawn in view of applicant's claim amendment filed 17 August 2007.

Page 3

## Drawings

4. The drawings are objected to because:

Fig. 1A, 1B, 2A, 2B are shown in the drawings. However, the specification only discusses Fig. 1 and Fig. 2 as a whole.

Fig. 6 shows two additional data points for "control" data set on August 10 and 17 above the "Test" data set. These data points are not discussed in the specification. In addition, the date increments on the x-axis are not proportionally and accurately represented.

Figs. 7(A) and 7(B) as discussed on lines 24 and 29 on page 13 of the specification are not properly labeled in Fig. 7.

On page 15, lines 15-20 of the instant specification teaches that Fig. 8 shows dissolved oxygen went from 0.5mg/l to 10.8 mg/l in nine minutes. However, Fig. 8 does not show dissolved oxygen levels over time. Instead, it shows temperature variation over time.

The claimed feature of a side arm flow portion, wherein the oxygen emitter reside, is not shown in any of the figures.

Corrected drawing sheets in compliance with 37 CFR 1.121(d) are required in reply to the Office action to avoid abandonment of the application. Any amended replacement drawing sheet should include all of the figures appearing on the immediate prior version of the sheet, even if only one figure is being amended. The figure or figure number of an amended drawing should not be labeled as "amended." If a drawing figure is to be canceled, the appropriate figure must be removed from the replacement sheet,

JA490

Page 4

and where necessary, the remaining figures must be renumbered and appropriate changes made to the brief description of the several views of the drawings for consistency. Additional replacement sheets may be necessary to show the renumbering of the remaining figures. Each drawing sheet submitted after the filing date of an application must be labeled in the top margin as either "Replacement Sheet" or "New Sheet" pursuant to 37 CFR 1.121(d). If the changes are not accepted by the examiner, the applicant will be notified and informed of any required corrective action in the next Office action. The objection to the drawings will not be held in abeyance.

# Priority

5. This application is a CIP of previously filed US patent application 10/372,017, now Patent No. 6,689,262. However, the parent patent does not disclose the claimed stabilizing hardware, the claimed water hose and the claimed hydroponic circulating system as recited in independent claims 1 and 25-26. Therefore, the instant application does NOT benefit from the effective filing date of the parent patent. The effective filing date of the current application is 10 December 2003.

## Claim Rejections - 35 USC § 102

6. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

7. Claims 1-3, 13, 15 and 17-22 are rejected under 35 U.S.C. 102(b) as being anticipated by Takesako et al. US 2002/0074237 A1(Takesako).

JA491

Page 5

Takesako teaches a water electrolyzer comprising a fluid conduit having a fluid inlet and a fluid outlet connected with a conduit lumen(Fig. 1(a)-(b), #1, 21, 22). Takesako also teaches an electrolysis cell positioned within the conduit lumen and parallel to a flow axis of the conduit lumen(Fig. 1(b), paragraph [0021]). The electrolysis cell as taught by Takesako comprises a plurality of matched sets of anodes and cathodes and secured to electrode connecting rods by conductive bolts and spacers(Figs. 2-3, #2, 4, 25-27 and 31-33, paragraph [0056]). In addition, the electrodes are expanded metal mesh(paragraphs [0012, 0062] and the distance between the electrodes does not exceed 3.0mm(paragraph [0017]). Takesako further teaches that the electrolysis cell in the conduit lumen is connected to a power source (Fig. 1(b)).

Regarding claims 1-3, 17-18 and 21, the water electrolyzer as taught by Takesako reads on the claimed flow through oxygenator. The electrolysis cell within the conduit lumen as taught by Takesako reads on the claimed oxygen emitter. The electrode connecting rods, the conductive bolts and the conductive spacers that secure the plurality of matched sets of electrodes as taught by Takesako reads on the claimed stabilizing hardware.

Regarding claim 13, based on the shape of the electrode connecting rods and the way the electrodes are structured and secured, the examiner takes a position that the power source in the apparatus of Takesako is inherently connected to the electrode connecting rods, which is a part of the claimed stabilizing hardware, to provide electricity to the electrodes.

JA492

Page 6

Regarding claim 15, Takesako further teaches that the polarity of the electrodes are reversed periodically(paragraphs [0011,0024, 0063-0065]). Therefore, the perforated electrodes proximate the conduit wall in the apparatus of Takesako function as anodes and the non-perforated electrodes proximate a conduit center in the apparatus of Takesako function as cathodes during periods of operation, which meets the limitation of the instant claim 15.

Regarding claim 19, Takesako further teaches a controller connected to a flow detecting circuit for controlling the voltage and the polarity applied to the water electrolysis cell(paragraphs [0063-0065]). Therefore, the controller as taught by Takesako is inherently capable of operating the power source in the claimed manner.

Regarding claims 20 and 22, since Takesako teaches a flow through water electrolyzer that is structurally the same as the claimed flow through oxygenator, the examiner takes a position that the apparatus of Takesako is capable of generating oxygen sufficient to form a supersaturated aqueous medium as claimed.

8. Claims 1-2, 13, 17 and 20-22 are rejected under 35 U.S.C. 102(b) as being anticipated by Hough et al. US 6,171,469 B1(Hough).

Hough teaches a water electrolyzer for increasing oxygen content of water (abstract, title), wherein the water electrolyzer comprises a flow conduit having an inlet and an outlet connected to the conduit lumen(Fig. 1 #11-12). Hough also teaches a plurality of matched sets of anodes and cathodes mounted to stabilizing hardware and positioned within the conduit lumen(Fig. 2C). The electrodes are connected to a power source(Fig. 1 #14, col. 3 lines 6-11). The electrodes in the water electrolyzer of Hough

JA493

Page 7

are metal(col. 3 lines 1-5) and are positioned parallel to the flow axis of the conduit(Fig. 2C).

Regarding claims, 1-2, 17 and 21, the water electrolyzer of Hough meets the structural limitations of the instant claims.

Regarding claim 13, based on the connection between the electrode plates and the stabilizing hardware, the examiner takes a position that the power source in the apparatus of Hough is inherently connected to the electrode connecting nuts and bolts and contacting wires(i.e. stabilizing hardware) to provide electricity to the electrodes.

Regarding claims 20 and 22, since Hough teaches a flow through water electrolyzer that is structurally the same as the claimed flow through oxygenator, the examiner takes a position that the apparatus of Hough is capable of generating oxygen sufficient to form a supersaturated aqueous medium as claimed.

# Claim Rejections - 35 USC § 103

9. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

10. Claims 4, 16 and 23-26 are rejected under 35 U.S.C. 103(a) as being

unpatentable over Takesako.

The teachings of Takesako are discussed in paragraph 7 above.

Regarding claim 4, the inter-electrode distance of not exceeding 3mm as taught

by Takesako encompasses the claimed gap of 0.045-0.060 inches(i.e. 1.143-1.524

JA494

Page 8

mm). Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed gap between electrodes from the disclosed gap of Takesako would have been obvious to one skilled in the art since Takesako teaches the same utilities in its disclosed inter-electrode distance.

Regarding claim 16, even though Takesako does not explicitly teach the claimed oxygen emitter positioned within a side arm flow portion of the conduit lumen, one of ordinary skill in the art would have found it obvious to have positioned the water electrolysis cell in any part of the conduit lumen, including the claimed side arm flow portion, with expected success since water flows through any part of the conduit lumen and the location of the electrolysis cell is an obvious variation absence any evidence that a specific location is superior.

Regarding claims 23-26, even though Takesako does not explicitly teach using the water electrolysis cell in the claimed watering hose or the claimed hydroponic circulating system, one of ordinary skill in the art would have found it obvious to have adapted the water electrolysis cell as taught by Takesako in any suitable applications wherein electrolyzed water is desirable, including the claimed watering hose and the claimed hydroponic circulating system.

11. Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Takesako, in view of Cairns et al. US 4,587,001(Cairns).

The teachings of Takesako are discussed in paragraphs 7 and 10 above. However, Takesako does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 9

Cairns teaches an cathode for use in an electrolytic cell(abstract). Cairns further teaches an titanium anode having a electro-catalytically active coating material comprising one or more oxides of platinum group metals such as platinum and iridium(col. 5 lines 15-25).

Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the anode of Cairns into the water electrolysis cell of Takesako as the anode since Cairns teaches that platinum group metal oxides is a good electrocatalytically active material for an anode of an electrolytic cell and the application of such coating on an anode is well known in the art(col. 5 lines 15-16 and 32-33).

12. Claims 3-4, 16, 18-19 and 23-26 are rejected under 35 U.S.C. 103(a) as being unpatentable over Hough, and further in view of Takesako.

The teachings of Hough are discussed in paragraph 8 above. However, Hough does not explicitly teach the claimed inter-electrode distance, the claimed metal mesh electrode and the claimed controller.

The teachings of Takesako are discussed in paragraphs 7 and 10 above.

Regarding claims 3-4, it would have been obvious to one of ordinary skill in the art to have incorporated the inter-electrode distance of not exceeding 3mm as taught by Takesako into the water electrolyzer of Hough in order to receive an increased current without using a very high voltage as taught by Takesako. In addition, the inter-electrode as taught by Hough in view of Takesako encompasses the claimed gap of 0.045-0.060 inches(i.e. 1.143-1.524 mm). Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed gap between electrodes from the disclosed

JA496

Page 10

gap of Hough in view of Takesako would have been obvious to one skilled in the art since Hough in view of Takesako teach the same utilities in their disclosed interelectrode distance.

Regarding claim 16, even though Hough in view of Takesako do not explicitly teach the claimed oxygen emitter positioned within a side arm flow portion of the conduit lumen, one of ordinary skill in the art would have found it obvious to have positioned the water electrolysis cell in any part of the conduit lumen, including the claimed side arm flow portion, with expected success since water flows through any part of the conduit lumen and the location of the electrolysis cell is an obvious variation absence any evidence that a specific location is superior.

Regarding claim 18, Takesako further teaches that perforated electrode plates facilitate the flow of water into the flow passages between the electrode plates (paragraph 0062). Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the perforated electrode plates as taught by Takesako into the water electrolyzer of Hough in order to facilitate the flow of water into the flow passages as taught by Takesako.

Regarding claim 19, Takesako further teaches the use of a control circuit and a flow detecting circuit to control the voltage from the power source applied to the electrolyzer(paragraphs[0063-0065]). Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the control circuit and the flow detecting circuit as taught by Takesako into the water electrolyzer of Hough in order to control the voltage of the electrolyzer as taught by Takesako.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 11

Regarding claims 23-26, even though Hough in view of Takesako do not explicitly teach using the water electrolysis cell in the claimed watering hose or the claimed hydroponic circulating system, one of ordinary skill in the art would have found it obvious to have adapted the water electrolyzer as taught by Hough in view of Takesako in any suitable applications wherein electrolyzed water is desirable, including the claimed watering hose and the claimed hydroponic circulating system.

13. Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Hough, in view of Cairns.

The teachings of Hough are discussed in paragraph 8 above.

However, Hough does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

The teachings of Cairns are discussed in paragraph 11 above.

Therefore, it would have been obvious to one of ordinary skill in the art to have

incorporated the anode of Cairns into the water electrolyzer of Hough as the anode

since Cairns teaches that platinum group metal oxides is a good electro-catalytically

active material for an anode of an electrolytic cell and the application of such coating on

an anode is well known in the art(col. 5 lines 15-16 and 32-33).

# **Double Patenting**

14. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., In re Berg, 140

JA498

Page 12

F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); In re Goodman, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); In re Longi, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); In re Van Ornum, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); In re Vogel, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and In re Thorington, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

15. Claims 1-4, 9, 13, 15 and 18-22 are rejected on the ground of nonstatutory

obviousness-type double patenting as being unpatentable over claims 1-6, 9 and 13-14

of U.S. Patent No. 6,689,262 B2(US'262) in view of Takesako.

Claims of U.S. Patent No. 6,689,262 B2 teach an oxygen emitter that is

structurally similar to the emitter of the claimed flow-through oxygenator.

However, claims of US'262 does not explicitly teach that the anodes and the

cathodes are mounted to stabilizing hardware.

The teachings of Takesako are discussed in paragraph 7 above. Therefore, it

would have been obvious to one of ordinary skill in the art to have adapted the electrode

connecting rods, the conductive bolts and spacers(i.e. stabilizing hardware) as taught

by Takesako into the oxygen emitter of US'262 in order to securely position the oxygen

emitter within a flow conduit as taught by Takesako.

Page 13

# Allowable Subject Matter

16. Claim 14 is objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims.

17. The following is a statement of reasons for the indication of allowable subject matter: The prior art of record does not teach or fairly suggest, either alone or in combination, the claimed flow through oxygenator comprising three matched sets of anodes and cathodes attached to stabilizing hardware in adjacent relation such that each matched set resides at a 120° angle to the adjacent matched sets.

# Response to Arguments

18. Applicant's arguments filed 17 August 2007 have been considered but are moot in view of the new ground(s) of rejection.

# Conclusion

19. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure.

Nishiki et al. US 5,015,354 teaches a bi-polar water electrolyzer comprising a water electrolysis cell positioned within a flow conduit and secured by stabilizing hardware, wherein the electrodes are parallel to the flow axis.

20. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

JA500

Page 14

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Lois Zheng whose telephone number is (571) 272-1248. The examiner can normally be reached on 8:30am - 5:00pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Roy King can be reached on (571) 272-1244. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 15

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

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				Lois 2	Zheng	1793	Page 1 of 1
				U.S. PATENT I	DOCUMENTS		
*		Document Number Country Code-Number-Kind Code	Date MM-YYYY		Name		Classification
*	Α	US-5,015,354	05-1991	Nishiki et al.			204/254
*	в	US-2002/0074237	06-2002	Takesako et a	l.		205/628
*	С	US-6,171,469	01-2001	Hough et al.			205/743
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\*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).) Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

Notice of References Cited

Part of Paper No. 20071029

	Search	Notes		Application/Control No. 10/732,326 Examiner		Patent under on AMES ANDR	
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S12	2	"5887383".pn.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 10:28
S11	1	10/732326	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 10:28
S1	12	("4252856" "5534143" "5982609" " 6315886" "6394429" "6689262"). PN.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:29
S13	2	"6689262".PN.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:30
S14	10	("3975269"   "4012319"   "4732661"   "4908109"   "5049252"   "5182014"   "5534143"   "6315886"   "6394429"   "6471873"   "WO 9521795").PN. OR ("6689262"). URPN.	US-PGPUB; USPAT; USOCR	OR	ON	2007/10/19 11:33
S16	17869	(tube pipe conduit) with (cell unit electrolyzer) and ((anode same cathode) electrode) with (gap distance apart spac\$3)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR .	ON	2007/10/19 11:36
S21	727	(tube pipe conduit) with oxygenat\$3 with water	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:45
S20	180	(tube pipe conduit) with ((oxygen "O.sub.2") near3 emitt\$3)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:4

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S29	3152	(tube pipe conduit hose cylind\$5) with oxygen\$5 and ("204" "205"). clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:47
S28	519	(tube pipe conduit hose cylind\$5) with oxygen\$5 with water and ("204" "205").clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:47
S27	27	(tube pipe conduit) with oxygenat\$3 with water and ("204" "205").clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:47
S26	11	(S18 not S17) and ("204" "205"). clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:47
S25	0	(tube pipe conduit hose cylind\$5) with oxygen\$5 and ("204" "205"). ccls.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:47
S30	27	S29 and (oxygenator superoxygen\$4 super adj oxygen\$4)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:49
S18	629	S15 and (oxygenator superoxygen\$4 super adj oxygen\$4)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:49
S15	435605	(tube pipe conduit) with (cell unit electrolyzer)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:55

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**OWT Ex. 2118** Tennant Company v. OWT IPR2021-00625

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S31	1603	(tube pipe conduit) with (cell unit electrolyzer) and ("204" "205").clas. and parallel\$3 with (anode cathode electrode)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 11:56
S33	12	("2829095"   "4252628"   "4402810"   "4413041"   "4734181"   "4755272").PN. OR ("5015354"). URPN.	US-PGPUB; USPAT; USOCR	OR	ON	2007/10/19 16:52
S32	322	S31 and (generat\$4 produc\$4 mak\$3) near3 (oxygen "O.sub.2")	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 17:07
534	1603	(tube pipe conduit) with (cell unit electrolyzer) and ("204" "205").clas. and parallel\$3 with (anode cathode electrode)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 17:08
S17	23	S16 and (oxygenator ' superoxygen\$4 super adj oxygen\$4)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 17:08
S35	3	S34 and (oxygenator superoxygen\$4 super adj oxygen\$4)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/19 17:09
S37	6478	flow adj through near2 (electrolyzer cell unit oxygenator) and (inch\$2 mm millimeter)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/23 15:05
S36	17718	flow adj through near2 (electrolyzer cell unit oxygenator)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/23 15:05

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S38	592	flow adj through near2 (electrolyzer cell unit oxygenator) and (inch\$2 mm millimeter) with (electrode anode cathode) and ("204" "205"). clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/10/23 15:10
S39	49	("2468357"   "2864750"   "3095365"   "3523891"   "3654119"   "3728245"   "3819504"   "3865710"   "3925176"   "3943044"   "4017375"   "4119517"   "4132620"   "4160716"   "4180445"   "4312736"   "4385973"   "4419206"   "445216"   "4436601"   "4451341"   "4528083"   "4572775"   "4623436"   "4639303"   "4761208"   "4781805"   "4783246"   "4784735"   "4790914"   "4797182"   "4839007"   "4917782"   "4936979"   "5062940"   "5292412"   "5324398"   "5328584"   "5389214"   "5427667"   "5460702"   "5728287"   "6171469").PN. OR ("6478949"). URPN.	US-PGPUB; USPAT; USOCR	OR	ON 、	2007/10/29 11:49

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JA509

### PATTERSON THUENTE SKAAR REGIVED 2002 CENTRAL FAX CENTER

### AUG 1 7 2007

Attorney Docket No.: 4056.02US01

### PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

James Andrew Senkiw

Application No.: 10/732,326

Confirmation No.: 7020

Group Art Unit: 1742

Examiner: Zheng, Lois L.

Filed: December 10, 2003

For: FLOW-THROUGH OXYGENATOR

#### AMENDMENT

Mail Stop Amendment Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

#### INTRODUCTORY COMMENTS

In response to the Office Action mailed May 24, 2007, amendment to the above-

identified patent application is requested.

The present amendment comprises the following sections:

- A. Amendments to the Claims
- B. Remarks

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

CERTIFICATE OF MAILING

I hereby certify that this document is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1459, Alexandrif, VA743, -1450 on

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### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 513 of 1320

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PATTERSON THUENTE SKAAR

AUG 1 7 2007

Application No. 10/732,326

### AMENDMENTS TO THE CLAIMS

1. (Currently Amended) A flow through oxygentaor consisting of comprising:

a fluid conduit having a fluid inlet and a fluid outlet fluidly connected with a conduit lumen;

an <u>oxygen</u> emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, <u>the oxygen emitter including a plurality of matched sets of anodes and cathodes wherein the matched sets of anodes and cathodes are mounted to stabilizing hardware such that the oxygen emitter is positioned within the conduit lumen comprising an anode separated at a critical distance from a cathode both within an aqueous medium and in aqueous communication with each other; and</u>

a power source all in electrical communication with each other, wherein the oxygen emitter is placed within or adjacent to a conduit for flowing water.

2. (Currently Amended) The flow through oxygenator emitter of claim 1, wherein the each anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the each cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

3. (Currently Amended) The <u>flow through oxygenator</u> eritical distance of claim 1<u>, wherein</u> the anode and cathode within each matched set are separated by a spacer such to maintain a gap of which is 0.005 to 0.140 inches between the anode and cathode.

4. (Currently Amended) The flow through oxygenator critical-distance of claim 1 3, wherein the gap which is 0.045 to 0.060 inches.

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PATTERSON THUENTE SKAAR

Ø 004

Application No. 10/732;326

5. (Withdrawn) The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

6. (Withdrawn) A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

9. (Currently Amended) The flow through oxygenator emitter of claim 1 wherein the each anode is platinum and iridium oxide on a support and the each cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

10. (Withdrawn) A method to increase the oxygen content of flowing water comprising passing flowing water through a conduit comprising the flow-through oxygenator of claim 1.

11. (Withdrawn) The method of claim 11 wherein the flowing water has a temperature of 1 to 40 degrees Celsius.

12. (Withdrawn) The method of claim 11 wherein the flowing water becomes supersaturated with oxygen.

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PATTERSON THUENTE SKAAR

Ø 005

Application No. 10/732,326

13. (New) The flow through oxygenator of claim 1, wherein the power source is electrically connected to the stabilizing hardware for powering the plurality of matched sets of anodes and cathodes.

14. (New) The flow through oxygenator of claim 1, wherein the plurality of matched sets comprises three matched sets of anodes and cathodes attached to the stabilizing hardware in adjacent relation such that each matched set resides at a 120° angle to the adjacent matched sets.

15. (New) The flow through oxygenator of claim 1, wherein the plurality of matched sets of anodes and cathodes are attached to the stabilizing hardware with the anodes proximate a conduit wall and the cathodes proximate a conduit center.

16. (New) The flow through oxygenator of claim 1, wherein the conduit lumen comprises a main flow portion and a side arm flow portion and wherein the oxygen emitter is positioned within the side arm flow portion using the stabilizing hardware.

17. (New) The flow through oxygenator of claim 1, wherein the plurality of matched sets of anodes and cathodes define plates positioned parallel to a flow axis of the conduit lumen.

18. (New) The flow through oxygenator of claim 1, wherein each cathode comprises a mesh screen.

19. (New) The flow through oxygenator of claim 1, further comprising:

a controller selectively operating the power source, such that the power source supplies power to the plurality of matched sets of anodes and cathodes when the aqueous

4

PAGE 5/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

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PATTERSON THUENTE SKAAR

Application No. 10/732,326

medium is flowing through the conduit lumen and withholds power when the aqueous medium is not flowing through the conduit lumen.

20. (New) The flow through oxygenator of claim 1, wherein the oxygen emitter is sized to generate oxygen sufficient to form a supersaturated aqueous medium.

21. (New) The flow through oxygenator of claim 1, wherein the aqueous medium is water.

22. (New) The flow through oxygenator of claim 21, wherein the oxygen emitter is sized to generate oxygen sufficient to form superoxygenated water.

23. (New) The flow through oxygenator of claim 1, wherein the fluid conduit is a watering hose.

24. (New) The flow through oxygenator of claim 1, wherein the fluid conduit is a hydroponic circulating system.

25. (New) A flow through oxygenator comprising:

a watering hose having a hose lumen; and

an oxygen emitter operably mounted within the hose lumen.

26. (New) A flow through oxygenator comprising:

a hydroponic circulating system having a circulating lumen; and an oxygen emitter operably mounted within the circulating lumen.

PAGE 6/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

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PATTERSON THUENTE SKAAR

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Application No. 10/732,326

### **REMARKS**

Claims 1-4 and 9-12 are pending. By this Amendment, claims 1-4 and 9 are amended, new claims 13-26 are added and claims 10-12 are withdrawn. Claims 5-8 have been previously withdrawn. Support for the amendments can be found in the application, figures and claims as originally filed and more specifically at Page 4, Lines 18-28 and Page 13, Line 22 – Page 15, Line 12 as well as Figure 7. No new matter is introduced by way of the present amendments.

#### Status of Claims

By way of the present amendment, claims 1-4, 9 and new claims 13-26 are presently pending with claims 5-8 and 10-12 being presently or previously withdrawn.

#### Election/Restrictions

Applicant respectfully acknowledges the constructive election of claims 1-4 and 9.1

#### Terminal Disclaimer

Claims 1-4 and 9 were previously rejected on the ground of nonstatutory obviousnesstype double patenting. Applicant respectfully asserts that the need for a Terminal Disclaimer to overcome a nonstatutory obviousness-type double patenting rejection has been overcome through the present amendment to independent claim 1 and the addition of new independent claims 25 and 26. As claims 1, 25 and 26 are patently distinct from claims 1-6 of U.S. Patent No. 6,689,262, Applicant respectfully requests said rejections be withdrawn.

PAGE 7/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

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### Ø 008

#### Application No. 10/732,326

### Claim Rejections - 35 USC §102

In the Office Action mailed May 24, 2007, claims 1-3 were rejected under 35 U.S.C. 102(e) as being anticipated by U.S. Patent No. 6,328,875 to Zappi et al. In response, Applicant presents amended claim 1 to further clarify the presently claimed invention. With the present response, Applicant has amended independent claim 1 to clarify the presently claimed flow through oxygenator as comprising an oxygen emitter positioned within a conduit lumen of a fluid conduit.

Zappi et al. discloses an electrolytic apparatus and related methods of use for the electropurification of contaminated aqueous media. Zappi et al. discloses the use of an electrolytic cell in an "open configuration" allowing for the controlled leakage of aqueous electrolyte solution and gaseous by-products (See Col. 4, Lines 9-43, Col. 5, Line 23 – Col. 6, Line 10, Col. 6, Lines 24-50 and Figures 1, 2 and 3). While Zappi et al. discusses the use of a conduit means (Col. 3, Lines 52-54) or pipe (Col. 14, Lines 23-37) for feeding aqueous electrolyte solution to the electrodes in the electrolyzer zone (Col. 3, Lines 52-54), Zappi et al. is absent any disclosure relative to the positioning of an oxygen emitter directly within the conduit lumen of a fluid conduit as presently claimed. Further evidence of Zappi et al.'s lack of disclosure relative to the positioning of an oxygen emitter within a conduit lumen of a fluid conduit as presently claimed. Further evidence of zappi et al.'s lack of disclosure relative to the positioning of an oxygen emitter within a conduit lumen of a fluid conduit as presently claimed. Further evidence of presently amended independent claim 1, Applicant respectfully requests said rejection be withdrawn.

PAGE 8/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

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#### Ø 009

Application No. 10/732,326

#### <u>Claim Rejections - 35 USC §103</u>

In the Office Action mailed May 24, 2007, claim 4 was rejected under 35 U.S.C. 103(a) as being unpatentable over Zappi . As discussed above, Zappi et al. fails to disclose an oxygen emitter positioned within a conduit lumen of a fluid conduit as presently claimed in independent claim 1. As such, Zappi et al. fails to establish a *prima facie* case of obviousness with respect to independent claim 1. Applicant respectfully requests said rejection be withdrawn.

In the Office Action mailed May 24, 2007, claim 9 was rejected under 35 U.S.C. 103(a) as being unpatentable over Zappi in view of U.S. Patent No. 4,587,001 to Cairns et al. As discussed above, Zappi et al. fails to disclose the positioning of an oxygen emitter within the conduit lumen of a fluid conduit. Cairns et al. is directed solely to a cathode having a metallic substrate and is absent any teaching, suggestion or motivation relative to the positioning of an oxygen emitter within the conduit lumen of a fluid conduit. As such, neither Zappi et al. nor Cairns et al., considered individually or combination, establish a *prima facie* case of obviousness with respect to presently amended independent claim 1. Applicant respectfully requests said rejection be withdrawn.

In the Office Action mailed May 24, 2007, claims 1-4 were rejected under 35 U.S.C. 103(a) as being unpatentable over U.S. Patent No. 4,225,401 to Divisek et al. As admitted in the Office Action mailed May 24, 2007, Divisek does not teach an electrolyzer placed directly within a conduit as presently claimed in amended independent claim 1. Contrary to the assertions within the office action, there is simply no support that would lead one of skill in the art, utilizing either the explicit disclosure of Divisek or simple "common sense" to position the electrolyzer of Divisek adjacent to a fluid conduit let alone within the fluid conduit as presently

### PAGE 9/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

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PATTERSON THUENTE SKAAR

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#### Application No. 10/732,326

claimed. The teachings of Divisek in which anode and cathode chambers are separated by a specified separator and preferred operation of the invention is conducted at temperatures of 300°C to 600°C could not possibly teach, suggest or motivate one of skill in the art to consider positioning the electrolyzer either adjacent to or directly within a fluid conduit as presently claimed within independent claim 1. As such, Divisek et al. fails to establish a case of *prima facie* obviousness with respect to presently amended independent claim 1. Applicant respectfully requests said rejection be withdrawn.

In the Office Action mailed May 24, 2007, claim 9 was rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek et al. in view of Cairns et al. As discussed previously, neither Divisek et al. nor Cairns et al., considered individually or in combination, teach or suggest the positioning of an oxygen emitter directly within a conduit lumen of a fluid conduit. As such, the proposed combination of Divisek et al. and Cairns et al. fails to establish a *prima facie* case of obviousness with respect to presently amended independent claim 1. Applicant respectfully requests said rejection be withdrawn.

#### New Claims

Newly added independent claims 25 and 26 each contain the structural limitation of an oxygen emitter being operably mounted within a conduit lumen of a conduit. As discussed previously with respect to the present rejections to independent claim 1, none of the presently cited art considered individually or in combination teaches the positioning of an oxygen emitter directly within a conduit lumen of a fluid conduit. As such, Applicant respectfully asserts that newly added independent claims 25 and 26 are in condition for allowance.

PAGE 10/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR: USPTO-EFXRF-2/4 \* DNIS: 2738300 \* CSID: 6123499266 \* DURATION (mm-ss): 03-56

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PATTERSON THUENTE SKAAR

Ø011

Application No. 10/732,326

In view of the foregoing, it is submitted that this application is in condition for allowance.

Favorable consideration and prompt allowance of the application are respectfully requested.

The Examiner is invited to telephone the undersigned if the Examiner believes it would be useful to advance prosecution.

Respectfully submitted J. Paul Haun Registration No. 53,003

Customer No. 24113 Patterson, Thuente, Skaar & Christensen, P.A. 4800 IDS Center 80 South 8th Street Minneapolis, Minnesota 55402-2100 Telephone: (612) 349-3009

PAGE 11/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR: USPTO-EFXRF-2/4 \* DNIS: 2738300 \* CSID: 6123499266 \* DURATION (mm-ss): 03-56

PATTERSON THUENTE SKAAR

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### AUG 1 7 2007

Attorney Docket No.: 4056.02US01

PATENT APPLICATION

Confirmation No.: 7020

Examiner: Zheng, Lois L.

Group Art Unit: 1742

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

James Andrew Senkiw

Application No.: 10/732,326

Filed: December 10, 2003

For: FLOW-THROUGH OXYGENATOR

#### SUBSTITUTION OF ATTORNEY

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

I hereby appoint the practitioners associated with Customer Number 24113 to prosecute

this application and to transact all business in the Patent and Trademark Office connected therewith.

Address all telephone calls to: J. Paul Haun at telephone number (612) 349-3009.

Address all correspondence to:

Customer Number 24113 J. Paul Haun Patterson, Thuente, Skaar & Christensen, P.A. 4800 IDS Center, 80 South 8th Street Minneapolis, Minnesota 55402-2100

Please grant any extension of time necessary for entry; charge any fee due to Deposit Account No. 16-0631.

CERTIFICATE OF MAILING

I hereby certify that this document is being deposite	ed with the United States Postal Service with sufficient postage as first class
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Date of Deposit	A. Paul Haun
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PATTERSON THUENTE SKAAR

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Application No. 10/732,326

Please reference Attorney Docket No. 4056.02US01 on all correspondence. Additionally, please charge any future fees to Deposit Account No. 16-0631.

All previous powers of attorney granted in this case are hereby revoked.

Aqua Innovations, Inc., Assignee

8-15-07 Date:

Signature

ÌSRM

Name Printed or Typed

(D)

Title

PAGE 13/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR: USPTO-EFXRF-2/4 \* DNIS: 2738300 \* CSID: 6123499266 \* DURATION (mm-ss): 03-56

**JA521** 

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PATTERSON THUENTE SKAAR

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PATENT APPLICATION

Confirmation No.: 7020

Examiner: Zheng, Lois L.

Group Art Unit: 1742

Attorney Docket No.: 4056.02US01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

James Andrew Senkiw

Application No.: 10/732,326

December 10, 2003

For: FLOW-THROUGH OXYGENATOR

#### CERTIFICATE UNDER 37 CFR § 3.73(b)

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Filed:

Aqua Innovations, Inc., a corporation, states that it is the assignce of the entire right, title and interest in the patent application identified above by virtue of either:

 A. [X] An assignment from the inventor(s) of the patent application identified above. The assignment was recorded in the Patent and Trademark Office at Reel 017998, Frame 0954, or for which a copy thereof is attached.

OR

 B. [] A chain of title from the inventor(s), of the patent application identified above, to the current assignee as shown below:

1. From \_\_\_\_\_ to \_\_\_\_\_

The document was recorded in the Patent and Trademark Office at Reel \_\_\_\_\_\_, Frame \_\_\_\_\_\_ or for which a copy thereof is attached.

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Application No. 10/732,326

2. From \_\_\_\_\_ to \_\_\_\_

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thereof is attached.

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[ ] Additional documents in the chain of title are listed on a supplemental sheet.

[ ] Copies of assignments or other documents in the chain of title are attached.

The undersigned (whose title is supplied below) is empowered to sign this statement on behalf of the assignee.

Title

8-15-07 Date:

Signature Kichard ISRUC Name Printed or Typed

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PAGE 15/15 \* RCVD AT 8/17/2007 2:45:28 PM [Eastern Daylight Time] \* SVR:USPTO-EFXRF-2/4 \* DNIS:2738300 \* CSID:6123499266 \* DURATION (mm-ss):03-56

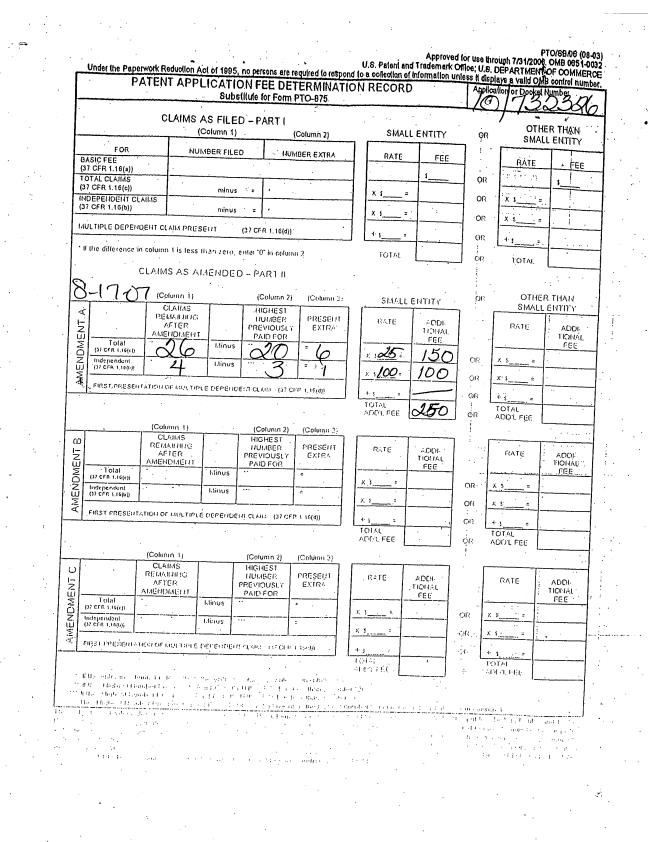
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### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 527 of 1320



OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 528 of 1320

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Part of Paper No. 20060620

JA526

# OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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Application Number	Application/Co	ntrol No.	Applicant(s)/Pa Reexamination	tent under			
	10/732,326		SENKIW, JAMES ANDREW				
Document Code - DISQ		Internal	nternal Document – DO NOT MAIL				

TERMINAL DISCLAIMER		
Date Filed : ユーユターᢧフ	This patent is subject to a Terminal Disclaimer	

Approved/Disapproved by:				
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U.S. Patent and Trademark Office

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003	James Andrew Senkiw	AQ1.002US1	7020
7590 05/24/2007 Kathleen R. Terry #314 1666 Coffman Street Falcon Heights, MN 55108		EXAMINER ZHENG, LOIS L		
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### Please find below and/or attached an Office communication concerning this application or proceeding.

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The time period for reply, if any, is set in the attached communication.

PTOL-90A (Rev. 04/07)

	Application No.	Applicant(s)				
1 2	10/732,326	SENKIW, JAMES ANDREW				
Office Action Summary	Examiner	Art Unit				
	Lois Zheng	1742				
The MAILING DATE of this communication appears on the cover sheet with the correspondence address Period for Reply						
<ul> <li>A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE <u>3</u> MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.</li> <li>Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.</li> <li>If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.</li> <li>Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).</li> </ul>						
Status		Í				
<ol> <li>Responsive to communication(s) filed on <u>27 March 2007</u>.</li> <li>This action is FINAL.</li> <li>2b)⊠ This action is non-final.</li> <li>Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under <i>Ex parte Quayle</i>, 1935 C.D. 11, 453 O.G. 213.</li> </ol>						
Disposition of Claims						
<ul> <li>4) Claim(s) <u>1-4 and 6-12</u> is/are pending in the appendix of the above claim(s) <u>6-8 and 10-12</u> is/are 5) Claim(s)</li></ul>	withdrawn from consideration. r election requirement. r. epted or b)□ objected to by the drawing(s) be held in abeyance. Se ion is required if the drawing(s) is ob	e 37 CFR 1.85(a). jected to. See 37 CFR 1.121(d).				
Priority under 35 U.S.C. § 119	· · · · · · · · · · · · · · · · · · ·					
<ul> <li>12) Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).</li> <li>a) All b) Some * c) None of: <ol> <li>Certified copies of the priority documents have been received.</li> <li>Certified copies of the priority documents have been received in Application No</li> </ol> </li> <li>3. Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).</li> <li>* See the attached detailed Office action for a list of the certified copies not received.</li> </ul>						
Attachment(s) 1) Notice of References Cited (PTO-892) 2) Notice of Draftsperson's Patent Drawing Review (PTO-948) 3) Information Disclosure Statement(s) (PTO/SB/08) Paper No(s)/Mail Date U.S. Patent and Trademark Office	4) Interview Summary Paper No(s)/Mail D 5) Notice of Informal F 6) Other:	ate				

PTOL-326 (Rev. 08-06)

Office Action Summary

Part of Paper No./Mail Date 20070522

Page 2

### DETAILED ACTION

### Continued Examination Under 37 CFR 1.114

1. A request for continued examination under 37 CFR 1.114, including the fee set forth in 37 CFR 1.17(e), was filed in this application after final rejection. Since this application is eligible for continued examination under 37 CFR 1.114, and the fee set forth in 37 CFR 1.17(e) has been timely paid, the finality of the previous Office action has been withdrawn pursuant to 37 CFR 1.114. Applicant's submission filed on 27 March 2007 has been entered.

### Status of Claims

2. Claim 1 is amended in view of the claim amendment filed 27 March 2007. New claims 10-12 are added in view of the claim amendment. Claims 6-8 remain withdrawn from consideration. Therefore, claims 1-4 and 9-12 are currently under examination.

Note, previously withdrawn claim 5 is missing in the claims filed 27 March 2007.

### Election/Restrictions

3. Newly submitted claims 10-12 are directed to an invention that is independent or distinct from the invention originally claimed for the following reasons:

New claims 10-12 and claims 1-3 and 9 are related as process and apparatus for its practice. The inventions are distinct if it can be shown that either: (1) the process as claimed can be practiced by another and materially different apparatus or by hand, or (2) the apparatus as claimed can be used to practice another and materially different process. (MPEP § 806.05(e)). In this case the apparatus as recited in claims 1-3 and 9

JA530

can be used to practice another and materially different process such as a process to produce hydrogen and oxygen.

Since applicant has received an action on the merits for the originally presented invention, this invention has been constructively elected by original presentation for prosecution on the merits. Accordingly, claims 10-12 are withdrawn from consideration as being directed to a non-elected invention. See 37 CFR 1.142(b) and MPEP § 821.03.

### Specification

4. The amendment to the specification filed on 27 March 2007 is entered.

### **Terminal Disclaimer**

5. The terminal disclaimer filed 28 February 2007 is improper because:

The application/patent being disclaimed has been improperly identified since the

number used to identify the application number 10/372,017 being disclaimed is

incorrect. The correct number is US Patent No. 6,689,262 B2.

### Status of Previous Rejections

6. The rejection of claims 1-4 and 9 under 35 U.S.C. 112, second paragraph, is

withdrawn in view of applicant's claim amendments filed 27 March 2007.

### Claim Rejections - 35 USC § 102

7. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that

form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(e) the invention was described in (1) an application for patent, published under section 122(b), by another filed in the United States before the invention by the applicant for patent or (2) a patent granted on an application for patent by another filed in the United States before the invention by the

JA531

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 3

applicant for patent, except that an international application filed under the treaty defined in section 351(a) shall have the effects for purposes of this subsection of an application filed in the United States only if the international application designated the United States and was published under Article 21(2) of such treaty in the English language.

8. Claims 1-3 are rejected under 35 U.S.C. 102(e) as being anticipated by Zappi et al. US 6,328,875 B1(Zappi).

Zappi teaches an electrolytic apparatus for electropurification of water(abstract), wherein the apparatus comprises a water feed, at least one cathode and at least one anode with inter-electrode gap between 0-2mm(Fig. 1 #12,18 and 20, col. 10, lines 13-15, col. 12 lines 45-49).

Regarding claims 1 and 3, Zappi teaches the generation of oxygen gas(Fig. 1). Therefore, the claimed electrolytic generation of microbubbles of oxygen inherently takes place when the electrolytic apparatus of Zappi is in operation. The inter-electrode distance of 0-2mm reads on the claimed critical distance from anode to cathode. Since the apparatus of Zappi is used to process water, the examiner concludes that the anode and the cathode in the apparatus of Zappi are both within an aqueous medium as claimed based on the broadest reasonable interpretation. In addition, Fig. 1 of Zappi further teaches that purified water drips from the electrode, which implies that the anode and the cathode as taught by Zappi are in aqueous communication with each other as claimed. The claimed power source is inherently present in the electrolytic apparatus of Zappi in order for it to be operational. Furthermore, the electrolytic apparatus as taught by Zappi is place adjacent to a conduit for flowing water(Fig. 1#22).

Regarding claim 2, Zappi further teaches that the anode and the cathode are a metal or metal oxide as claimed.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA532

Page 4

Page 5

Therefore, Zappi electrolytic apparatus anticipates the claimed flow-through

oxygenator and the claimed emitter.

### Claim Rejections - 35 USC § 103

9. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all

obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

10. Claim 4 is rejected under 35 U.S.C. 103(a) as being unpatentable over Zappi.

The teachings of Zappi are discussed in paragraph 8 above.

Regarding claim 4, the distance of 0-2mm between the electrodes as taught by

Zappi encompasses the claimed critical distance of 0.045 to 0.060 inches.

Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed critical distance from the disclosed range of Zappi would have been obvious to one skilled in the art since Zappi teaches the same utilities in its' disclosed critical distance range.

11. Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Zappi in view of Cairns et al. US 4,587,001(Cairns).

The teachings of Zappi are discussed in paragraph 8 above.

However, Zappi does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

Cairns teaches an cathode for use in an electrolytic cell(abstract). Cairns further teaches an titanium anode having a electro-catalytically active coating material

JA533

Page 6

comprising one or more oxides of platinum group metals such as platinum and iridium(col. 5 lines 15-25).

Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the anode of Cairns into the electrolyzer of Zappi as the anode since Cairns teaches that platinum group metal oxides is a good electro-catalytically active material for an anode of an electrolytic cell and the application of such coating on an anode is well known in the art(col. 5 lines 15-16 and 32-33).

12. Claims 1-4 are rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek et al. US 4,225,401(Divisek).

Divisek teaches a water electrolyzer for generating hydrogen and oxygen(abstract). The water electrolyzer comprises and anode separated at a distance from a cathode(Fig. 1), wherein both the anode and the cathode are within an aqueous medium as claimed. Divisek further teaches that the distance between the electrodes is about 1-3 mm(col. 3 lines 54-61).

Regarding instant claims 1 and 3, since the water electrolyzer of Divisek produces oxygen, the claimed oxygen microbubbles is inherently electrolytically generated when Divisek's water electrolyzer is in operation. In addition, Divisek teaches the claimed anode and cathode separated about 1-3 mm apart from each other, which reads on the claimed critical distance as recited in instant claim 3. The claimed power source is also inherently present in the water electrolyzer of Divisek. Furthermore, Divisek further teaches transfer of electrolyte from cathode chamber to anode chamber takes place in order to equalize the mass balance(col. 3 lines 47-50).

> OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA534

Therefore, the anode and the cathode are in aqueous communication with each other in the apparatus of Divisek.

Even though Divisek does not explicitly teach that its electrolyzer is place within or adjacent to a conduit for flowing water, one of ordinary skill in the art would have found the position of Divisek's electrolyzer at least adjacent to a water conduit obvious since water is added/fed to Devisek's electrolyzer for electrolysis to take place.

Therefore, the claimed flow-through oxygenator and the claimed emitter do not structurally distinguish from the water electrolyzer of Divisek.

Regarding claim 2, Divisek further teaches that the anode and the cathode are made of nickel(col. 4 lines 37-39), which meets the limitation of claimed metal anode and metal cathode.

Regarding claim 4, the distance of 1-3mm between the electrodes as taught by Divisek encompasses the claimed critical distance of 0.045 to 0.060 inches.

Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed critical distance from the disclosed range of Divisek would have been obvious to one skilled in the art since Divisek teaches the same utilities in its' disclosed critical distance range.

13. Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek in view of Cairns et al. US 4,587,001(Cairns).

The teachings of Divisek are discussed in paragraph 12 above.

However, Divisek does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 7

Page 8

Cairns teaches an cathode for use in an electrolytic cell(abstract). Cairns further teaches an titanium anode having a electro-catalytically active coating material comprising one or more oxides of platinum group metals such as platinum and iridium(col. 5 lines 15-25).

Therefore, it would have been obvious to one of ordinary skill in the art to have

incorporated the anode of Cairns into the electrolyzer of Divisek as the anode since

Cairns teaches that platinum group metal oxides is a good electro-catalytically active

material for an anode of an electrolytic cell and the application of such coating on an

anode is well known in the art(col. 5 lines 15-16 and 32-33).

### Double Patenting

14. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., In re Berg, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); In re Goodman, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); In re Longi, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); In re Van Ornum, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); In re Vogel, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and In re Thorington, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

Page 9

15. Claims 1-4 and 9 are rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-6 of U.S. Patent No. 6,689,262 B2. Although the conflicting claims are not identical, they are not patentably distinct from each other because the emitter of U.S. Patent No. 6,689,262 B2 is structurally the same as the emitter of the claimed flow-through oxygenator. Even though U.S. Patent No. 6,689,262 B2 does not explicitly teach the claimed flow through oxygenator, one of ordinary skill in the art would have found it obvious to use the instant emitter in an oxygenator as claimed since the emitter produces oxygen.

### **Response to Arguments**

16. Applicant's arguments filed 27 March have been fully considered but they are not persuasive.

In the remarks, applicant argues that Divisek teaches the use of a separator for separating the anode and the cathode into anode and cathode chambers. This separator is not present in the instant invention.

The examiner does not applicant's argument persuasive since the instant claim 1 uses open-ended transitional phase "comprising", which allows the presence of additional structural components in the claimed emitter, such as the separator as taught by Divisek.

Applicant further argues that Divisek does not teach that the water electrolyzer is placed within a conduit for flowing water.

The examiner does not find applicant's argument persuasive. As stated in paragraph 12 above, even though Divisek does not explicitly teach that its electrolyzer

JA537

Page 10

is place within or adjacent to a conduit for flowing water, one of ordinary skill in the art would have found the position of Divisek's electrolyzer at least adjacent to a water conduit obvious since water is added/fed to Devisek's electrolyzer for electrolysis to take place.

Applicant further argues that cathode is not located in an aqueous medium since the operating temperature as taught by Divisek is in the range of 300-600C. Therefore, any water would be in vapor form not in liquid form. The examiner does not find applicant's argument persuasive since the phase of water electrolyte depends upon the electrolysis operating temperature, therefore, is directed to a process limitation. As stated in MPEP 2114 [R-1], it is well settled that the manner in which a claimed apparatus is intended to be employed does not differentiate the claimed apparatus from a prior art apparatus as long as the prior art apparatus teaches all the structural limitations of the claim. Ex parte Masham, 2 USPQ2d 1647 (Bd. Pat. App. & Inter. 1987). In this case, since the apparatus as taught by Divisek is substantially the same structurally as the claimed apparatus, the examiner concludes that the rejection is proper.

Applicant's arguments with respect to claims 2, 4 and 9 are not found convincing since they are depended upon the non-convincing arguments of claim 1 above.

### Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Lois Zheng whose telephone number is (571) 272-1248. The examiner can normally be reached on 8:30am - 5:00pm.

JA538

Page 11

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Roy King can be reached on (571) 272-1244. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

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U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

Notice of References Cited

Part of Paper No. 20070522

	Search	Notes		Application/Control No.	SENK	ant(s)/Paten mination IW, JAMES	
				Examiner Lois Zheng	Art Unit 1742		
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L1	12	("4252856" "5534143" "5982609" " 6315886" "6394429" "6689262"). PN.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 15:18
L2	7	james near2 senkiw	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 15:19
L5	19	4 not 2	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 15:20
L3	390	(micro adj bubble microbubble) with oxygen	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 15:20
L4	24	3 and anode and cathode	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	<b>ON</b>	2007/05/22 15:23
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L7	744	6 and ("204" "205").clas.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 15:26
L9	1	"4048047".pn.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 17:10

## **EAST Search History**

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L8	282	6 and ("204" "205").clas. and (electrolytic electrolysis electrolyz\$4) near3 water	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 17:11
L10	15	8 and anode with (platinum Pt) with (iridium near2 oxide "IrO.sub.2")	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2007/05/22 17:12

## **EAST Search History**

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Page 2

JA543



## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.10/732,326ApplicantJames A. Senkiw.Filed12/10/2003Art Unit1742ExaminerLois L. Zheng

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

## RESPONSE AND AMENDMENT AFTER FINAL REJECTION

Dear Ms. Zheng:

Enclosed please find a response to the Office action of 02-05-2007. Please enter the Terminal Disclaimer over a Provisional Patent Application filed on February 24, 2007

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 5 of this paper.

1

**JA544** 

Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

#### Amendments to the Specification:

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Please replace the RELATED APPLICATIONS on page 1 of this application with the following:

This application is a continuation-in-part of United States Patent Application Number 10/372,017, filed on February 21, 2003, now United States Patent Number 6,689,262, issued February 10, 2004, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

2

Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

#### Amendments to the Claims:

This listing of the claims will replace all prior versions, and listings, of claims in the application.

#### Listing of Claims

Claim 1. (Currently amended) A flow-through oxygenator <u>consisting of comprising</u> an emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, comprising an anode separated at a critical distance from a cathode <u>both</u> within an aqueous medium <u>and in aqueous communication with each other</u>, and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. (Previously presented) The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. (Original) The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 4. (Original) The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 6. (Withdrawn) A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

Claim 7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

Claim 8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

Claim 9. (Previously presented) The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 10. (New) A method to increase the oxygen content of flowing water comprising passing flowing water through a conduit comprising the flow-through oxygenator of claim 1.

3

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 549 of 1320

Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

Claim 11. (New) The method of claim 11 wherein the flowing water has a temperature of 1 to 40 degrees Celsius.

Claim 12. (New) The method of claim 11 wherein the flowing water becomes supersaturated with oxygen.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

#### **REMARKS/ARGUMENTS**

The specification has been amended to correct the error in the response to the Office Action of 9 November 2006, to properly place the update of status of related applications on page 1 of the specification. New claims 10-12 are presented.

The Examiner has rejected claims 1, and claims 2-4 and 9 which depend on claim 1, under 35 U.S.C.§ 112, second paragraph, as being indefinite for not clearly pointing out that both the cathode and the anode are in an aqueous medium. Claim 1 as amended now recites that both anode and cathode are in an aqueous medium, and are in aqueous communication with each other. Applicant believes that amended claim 1 and the claims dependant on claim 1 satisfy 35 U.S.C. § 112, second paragraph.

The Examiner has rejected pending claims 1 and 3 under U.S.C. § 102(b) as being anticipated by Divisek et al. US 4,225,401 ("Divisek"). Applicant respectfully disagrees. Claim 1 has been amended to emphasize that unlike Divisek, the electrodes are not separated by a separator, but are both in aqueous medium and in aqueous communication with each other. It can be seen in Figure 1 that Divisek places his anode and cathode in two separate chambers. The abstract states in lines 4-5 that the anode and cathode chambers are separated from one another by a separator, which prevents aqueous communication. See also claim 1 of Divisek.

The Examiner further states that Figure 1 of Divisek shows that the "water electrolyzer" is placed within a conduit for flowing water. Applicant disagrees with that reading of Divisek. Figure 1, the abstract and all the examples of Divisek disclose that the electrolysis cell is static, that in, placed in two chambers, not a conduit. Furthermore, the Examiner is requested to take notice that water does not flow at the temperatures taught by Divisek, that is, 300 to 600 degrees Celsius, at which temperature  $H_2O$  exists as water vapor, not water. See Compact Edition of the Oxford English Dictionary (Oxford University Press, Oxford, England, 1971): "water- the *liquid* of which seas, lakes and rivers are composed." (Emphasis added.)

The Examiner further states that Divisek discloses a cathode located in an aqueous medium. Applicant respectfully disagrees. As stated above, Divisek does not disclose the presence of water, a liquid, which does not exist at the temperatures he discloses, but discloses water vapor, the gaseous, not the liquid, phase of  $H_2O$  (column 3, lines 2-4 and column 4, lines 56-58). His cathode is therefore not in an aqueous medium. Turning again to the Oxford English Dictionary, aqueous is defined as "1. of, or pertaining to, the nature of water; watery..." Also noted is the recitation on page 14, lines 10-12 of the specification that the present invention is operated at ambient temperature, that is, 10 to 12 degrees Celsius, at which temperature the substrate of Divisek would be a solid matter. This limitation is found in new claim 11.

**JA548** 

### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 551 of 1320

Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

The Examiner states that Divisek teaches the composition of the anode and cathode, thereby meeting the limitations of claim 2. Claim 2 is dependent on claim 1, which Applicant believes is patentable over Divisek. Divisek does not include the limitations of claim 1 and therefore does not anticipate claim 2.

The Examiner has rejected claim 4 under U.S.C. §103 (a) as being obvious from Divisek. Divisek is actually silent as to the distance between electrodes in his working examples 1 to 3. On column 3, lines 57-61, Divisek states that "distance between the electrodes which merely corresponds to the thickness of the separator are possible, in other words, for all practical purposes, this distance may amount to about 1-3 mm." While 1-3 mm overlaps with the critical distance recited in claim 4, claim 4 depends on claim 1 and includes all the limitations of claim 1, since claim1 has now been distinguished from Divisek, it is submitted that the rejection of claim 4 now longer applies.

The Examiner has rejected claim 9 under 35 U.S.C. § 103 (a) as being obvious from Divisek in view of Cairns et al U.S. 4,587,001. Claim 9 being dependant from claim 1, it should be read with all the limitationss of claim 1. The invention to be operative is not dependant on any specific anodes and cathodes (specification, page 4, line 1-8) but the platinum and iridium electrodes are more durable and thence comprise the best mode of making the invention. Applicant believes that claim 9 is now allowable.

The Examiner has rejected claim 1-4 on the ground of non-statutory obviousness-type double patenting over claims 1-6 of U.S. Patent 6,689,262B2. The Examiner points out that the open-ended transitional term "comprising" can include Divisek's separator. Claim 1 as amended now reads "consisting of" a closed-ended term that does not allow the inclusion of Divisek's separator. Applicant has submitted a Terminal Disclaimer which obviates this rejection

New claims 10-12 are presented to illustrate to the public how the invention is to be practiced. Support for these claims can be found in the specification, page 6, line15-16 and page 14, lines 10-12 and line 21. Page 14, lines 10-12 described the flowing water at ambient temperature, that is about 10 to 12 degrees Celsius. On page 14, line 21, the flowing water is described as supersaturated and milky with dissolved oxygen. Supersaturated is defined on page 6, line 15-16.

6

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 552 of 1320

Appl. No. 10/732,326 Amendment dated March 21, 2007 To Final Office Action of February 5, 2007

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The claims being amended to more distinctly claim the invention, Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Respectfully submitted, Applicant James A. Senkiw, by his Attorney,

Withlein K eny

Kathleen R. Terry Reg. No. 31884 (651) 659-9819 Krterry@visi.com

Please direct all correspondence to: Kathleen R. Terry 1666 Coffman Street #314 Falcon Heights, MN 55108

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 553 of 1320

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Continued Examination (RCE)	First Named Inventor	James Andr	ew Senkiw							
Transmittal		1742								
Mail Stop RCE	Art Unit	Lois Zheng								
Commissioner for Patents P.O. Box 1450	Examiner Name									
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This is a Request for Continued Examination (RCE) Request for Continued Examination (RCE) practice under 37 C	under 37 CFR 1.114 of the CFR 1 114 does not apply to any	above-identif	ied application.							
1995, or to any design application. See Instruction Sheet for R	CEs (not to be submitted to the	USPTO) on page	2.							
1. Submission required under 37 CFR 1.114 No	ote: If the RCE is proper, any pr	eviously filed une	entered amendments and							
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D / . A SIGNATURE OF APALIC.	ANT, ATTORNEY, OR AGENT	REQUIRED								
Signature Kathlein Killy	ny I	Date	21 March 2007							
Name (Print/Type) Kathleen R. Terry		Registration No.	31,884							
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I hereby certify that this correspondence is being deposited with the Uni addressed to: Mail Stop RCE, Corpmissioner for Patents, P. O. Box 145	ited States Postal Service with suffic i0, Alexandria, VA 22313-1450 or fa	ient postage as first csimile transmitted	t class mail in an envelope to the U.S. Patent and Trademark							
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including gathering, preparing, and submitting the complete application form to the USP10. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mall Stop RCE, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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		0		Examiner Name	e Lois	Zheng	
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This collection of information is required by 37 CFR 1.136. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 555 of 1320

KATHLEEN R. TERRY 1666 COFEMAN STREET, #314 FALCON HEIGHTS, MN 55108 22:70/980 21 Mar 20 07 \$ 3 %-JA A Cont DOLLARS 1-12-PARK MIDWAY BANK FOR AQLOODUST KCP

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 556 of 1320

MAR 2 7 2007 Junder the Second Redu	ction Act of 1995. no person	s are required to respond to a col	atent and Trademai	rk Office;	PTO/SB/21 (09-06 e through 03/31/2007. OMB 0651-003 U.S. DEPARTMENT OF COMMERC L displays a valid OMB control numbe
(to be used for all correspond Total Number of Pages in This	M lence after initial filing)	Application Number Filing Date First Named Inventor Art Unit Examiner Name Attorney Docket Number	10/732,326 12-03-2003 James Andrew Se 1742 Lois Zheng AQI.002US1	nkiw	
✓       Fee Transmittal Form         ✓       Fee Attached         ✓       Fee Attached         ✓       Amendment/Reply         ✓       After Final         △       After Final         △       After Final         △       After Final         △       After Final         ○       Extension of Time Re         □       Express Abandonmer         □       Information Disclosure         ○       Certified Copy of Prior         □       Certified Copy of Prior         □       Reply to Missing Parts:         □       Normal Parts:         □       Reply to Missing Parts:         □       Normal Parts:         □       Normal Parts:         □       Normal Parts:	aration(s)	Drawing(s) Licensing-related Papers Petition Petition to Convert to a Provisional Application Power of Attorney, Revocation Change of Correspondence A Terminal Disclaimer Request for Refund CD, Number of CD(s) Landscape Table on CD ks	ddress	Appea of App Appea (Appe Propri	Allowance Communication to TC al Communication to Board peals and Interferences al Communication to TC al Notice, Brief, Reply Brief) ietary Information s Letter Enclosure(s) (please Identify ):
Firm Name Kathleen R Signature Kathleen R Printed name Kathleen R Date 21 March 2	. Terry . Terry 007 CERTIFIC	CATE OF TRANSMISS	Reg. No. 31884 ON/MAILING	th the Ur	nited States Postal Service with Alexandria, VA 22313-1450 on
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This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to hile (and by the USP10 to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USP10. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 557 of 1320

	ATENT APPL	Substitute f					Docket Number 32,326	12/10/2003		To be Mailed
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	FOR		(Column	·	(Column 2)			OR		
T	BASIC FEE				NUMBER EXTRA	RATE (\$)	FEE (\$)	1	RATE (\$)	FEE (\$)
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**OWT Ex. 2118** Tennant Company v. OWT IPR2021-00625

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## UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office Address: COMMISSIONER FOR PATENTS P.O. Box 1450 Alexandria, Virginia 22313-1450 www.uspio.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003	AQ1.002US1	7020	
Kathleen R. Te	7590 03/15/2007		EXAM	INER
#314	•		ZHENG,	LOIS L
1666 Coffman Falcon Heights			ART UNIT	PAPER NUMBER
5	,		1742	
			MAIL DATE	DELIVERY MODE
			03/15/2007	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

PTOL-90A (Rev. 10/06)

	Application No.	Applicant(s)	
Advisory Action	10/732,326	SENKIW, JAMES	ANDREW
Before the Filing of an Appeal Brief	Examiner	Art Unit	T
	Lois Zheng	1742	
The MAILING DATE of this communication app	ears on the cover sheet v	vith the correspondence an	dress
HE REPLY FILED 28 February 2007 FAILS TO PLACE THIS			
<ul> <li>The reply was filed after a final rejection, but prior to or of this application, applicant must timely file one of the folloplaces the application in condition for allowance; (2) a N a Request for Continued Examination (RCE) in compliant time periods:</li> <li>a) The period for reply expiresmonths from the mailing by The period for reply expires on: (1) the mailing date of this no event, however, will the statutory period for reply expired</li> </ul>	owing replies: (1) an amend lotice of Appeal (with appea nce with 37 CFR 1.114. The ing date of the final rejection. Advisory Action, or (2) the dat	Iment, affidavit, or other evide al fee) in compliance with 37 e reply must be filed within or e set forth in the final rejection, v	ence, which CFR 41.31; or ( ie of the followin whichever is later.
Examiner Note: If box 1 is checked, check either box (a) o TWO MONTHS OF THE FINAL REJECTION. See MPEP	706.07(f).		
xtensions of time may be obtained under 37 CFR 1.136(a). The dat ave been filed is the date for purposes of determining the period of e nder 37 CFR 1.17(a) is calculated from: (1) the expiration date of the et forth in (b) above, if checked. Any reply received by the Office lat ay reduce any earned patent term adjustment. See 37 CFR 1.704( OTICE OF APPEAL	extension and the correspondin e shortened statutory period for er than three months after the	g amount of the fee. The approp reply originally set in the final O	oriate extension fe ffice action; or (2)
The Notice of Appeal was filed on A brief in con filing the Notice of Appeal (37 CFR 41.37(a)), or any ext a Notice of Appeal has been filed, any reply must be file <u>MENDMENTS</u>	tension thereof (37 CFR 41 ed within the time period set	37(e)), to avoid dismissal of forth in 37 CFR 41.37(a).	the appeal. Sind
<ul> <li>☑ The proposed amendment(s) filed after a final rejection</li> <li>(a) ☑ They raise new issues that would require further c</li> <li>(b) □ They raise the issue of new matter (see NOTE be</li> <li>(c) ☑ They are not decoded to place the application in h</li> </ul>	onsideration and/or search low);	(see NOTE below);	
<ul> <li>(c) ⊠ They are not deemed to place the application in b appeal; and/or</li> <li>(d) ☐ They present additional claims without canceling a</li> </ul>			g the issues for
NOTE: See Continuation Sheet. (See 37 CFR 1.	· //		
The amendments are not in compliance with 37 CFR 1.		of Non-Compliant Amendmen	t (PTOL-324).
<ul> <li>Applicant's reply has overcome the following rejection()</li> <li>Newly proposed or amended claim(s) would be</li> </ul>	<i>,</i>	separate, timely filed amendr	nent canceling t
non-allowable claim(s). For purposes of appeal, the proposed amendment(s): a	) 🛛 will not be entered, or	b) 🔲 will be entered and an	_
how the new or amended claims would be rejected is pr The status of the claim(s) is (or will be) as follows: Claim(s) allowed:	ovided below of appended.		
Claim(s) objected to: Claim(s) rejected: <i>1-4 and 9.</i>			
Claim(s) withdrawn from consideration: FFIDAVIT OR OTHER EVIDENCE			
<ul> <li>The affidavit or other evidence filed after a final action, because applicant failed to provide a showing of good a was not earlier presented. See 37 CFR 1.116(e).</li> </ul>	out before or on the date of Ind sufficient reasons why t	filing a Notice of Appeal will he affidavit or other evidence	not be entered is necessary a
The affidavit or other evidence filed after the date of filin entered because the affidavit or other evidence failed to showing a good and sufficient reasons why it is necessary	overcome <u>all</u> rejections un ary and was not earlier pres	der appeal and/or appellant f ented. See 37 CFR 41.33(d	ails to provide a
D. ☐ The affidavit or other evidence is entered. An explanatic EQUEST FOR RECONSIDERATION/OTHER		·	
<ol> <li>The request for reconsideration has been considered to See Continuation Sheet.</li> <li>Note the attached Information Disclosure Statement(a)</li> </ol>			ance because:
2. □ Note the attached Information Disclosure Statement(s) 3. □ Other:	. (F 10/36/06) Paper NO(S)	·	

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 560 of 1320

Continuation Sheet (PTO-303)

Application No. 10/732,326

Continuation of 3. NOTE: The new claim amendments change the scope of the finally rejected claims, therefore, require further search and consideration.

Continuation of 11. does NOT place the application in condition for allowance because: Applicant's argument are based on newly amended claims that change the scope of the finally rejected claims. The new claim amendments require further search and consideration.

<u>\_1</u> ROY KING JUNGAVISORY PATENT EXAMINER

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#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. Applicant Filed Art Unit Examiner

10/732,326 James A. Senkiw. 12/10/2003 1742 Lois L. Zheng

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

#### **RESPONSE AND AMENDMENT AFTER FINAL REJECTION**

Dear Ms. Zheng:

Enclosed please find a response to the Office action of 02-05-2007.

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 4 of this paper.

Not Entered

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.10/732,326ApplicantJames A. Senkiw.Filed12/10/2003Art Unit1742ExaminerLois L. Zheng

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

## **RESPONSE AND AMENDMENT AFTER FINAL REJECTION**

Dear Ms. Zheng:

Enclosed please find a response to the Office action of 02-05-2007.

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 4 of this paper.

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 563 of 1320

Appl. No. 10/732,326 Amendment dated February 24, 2007 To Final Office Action of February 5, 2007

#### Amendments to the Specification:

Please replace the RELATED APPLICATIONS on page 1 of this application with the following:

This application is a continuation-in-part of United States Patent Application Number 10/372,017, filed on February 21, 2003, now United States Patent Number 6,689,262, issued February 10, 2004, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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Appl. No. 10/732,326 Amendment dated February 24, 2007 To Final Office Action of February 5, 2007

#### Amendments to the Claims:

This listing of the claims will replace all prior versions, and listings, of claims in the application.

### Listing of Claims

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Claim 1. (Currently amended) A flow-through oxygenator <u>consisting of comprising</u> an emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, comprising an anode separated at a critical distance from a cathode <u>both</u> within an aqueous medium <u>and in aqueous communication with each other</u>, and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. (Previously presented) The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. (Original) The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 4. (Original) The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 6. (Withdrawn) A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

Claim 7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

Claim 8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

Claim 9. (Previously presented) The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

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Appl. No. 10/732,326 Amendment dated February 24, 2007 To Final Office Action of February 5, 2007

#### **REMARKS/ARGUMENTS**

The specification has been amended to correct the error in the response to the Office Action of 9 November 2006, to properly place the update of status of related applications on page 1 of the specification.

The Examiner has rejected claims 1, and claims 2-4 and 9 which depend on claim 1, under 35 U.S.C.§ 112, second paragraph, as being indefinite for not clearly pointing out that both the cathode and the anode are in an aqueous medium. Claim 1 as amended now recites that both anode and cathode are in an aqueous medium, and are in aqueous communication with each other. Applicant believes that amended claim 1 and the claims dependant on claim 1 satisfy 35 U.S.C. § 112, second paragraph.

The Examiner has rejected pending claims 1 and 3 under U.S.C. § 102(b) as being anticipated by Divisek et al. US 4,225,401 ("Divisek"). Applicant respectfully disagrees. Claim 1 has been amended to emphasize that unlike Divisek, the electrodes are not separated by a separator, but are both in aqueous medium and in aqueous communication with each other. It can be seen in Figure 1 that Divisek places his anode and cathode in two separate chambers. The abstract states in lines 4-5 that the anode and cathode chambers are separated from one another by a separator, which prevents aqueous communication. See also claim 1 of Divisek.

The Examiner further states that Figure 1 of Divisek shows that the "water electrolyzer" is placed within a conduit for flowing water. Applicant disagrees with that reading of Divisek. Figure 1, the abstract and all the examples of Divisek disclose that the electrolysis cell is static, that in, placed in two chambers, not a conduit. Furthermore, the Examiner is requested to take notice that water does not flow at the temperatures taught by Divisek, that is, 300 to 600 degrees Celsius, at which temperature  $H_2O$  exists as water vapor, not water. See Compact Edition of the Oxford English Dictionary (Oxford University Press, Oxford, England, 1971): "water- the *liquid* of which seas, lakes and rivers are composed." (Emphasis added.)

The Examiner further states that Divisek discloses a cathode located in an aqueous medium. Applicant respectfully disagrees. As stated above, Divisek does not disclose the presence of water, a liquid, which does not exist at the temperatures he discloses, but discloses water vapor, the gaseous, not the liquid, phase of  $H_2O$  (column 3, lines 2-4 and column 4, lines 56-58). His cathode is therefore not in an aqueous medium. Turning again to the Oxford English Dictionary, aqueous is defined as "1. of, or pertaining to, the nature of water; watery..."

The Examiner states that Divisek teaches the composition of the anode and cathode, thereby meeting the limitations of claim 2. Claim 2 is dependent on claim 1, which Applicant

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believes is patentable over Divisek. Divisek does not include the limitations of claim 1 and therefore does not anticipate claim 2.

The Examiner has rejected claim 4 under U.S.C. §103 (a) as being obvious from Divisek. Divisek is actually silent as to the distance between electrodes in his working examples 1 to 3. On column 3, lines 57-61, Divisek states that "distance between the electrodes which merely corresponds to the thickness of the separator are possible, in other words, for all practical purposes, this distance may amount to about 1-3 mm." While 1-3 mm overlaps with the critical distance recited in claim 4, claim 4 depends on claim 1 and includes all the limitations of claim 1, since claim1 has now been distinguished from Divisek, it is submitted that the rejection of claim 4 now longer applies.

The Examiner has rejected claim 9 under 35 U.S.C. § 103 (a) as being obvious from Divisek in view of Cairns et al U.S. 4,587,001. Claim 9 being dependant from claim 1, it should be read with all the limitationss of claim 1. The invention to be operative is not dependant on any specific anodes and cathodes (specification, page 4, line 1-8) but the platinum and iridium electrodes are more durable and thence comprise the best mode of making the invention. Applicant believes that claim 9 is now allowable.

The Examiner has rejected claim 1-4 on the ground of non-statutory obviousness-type double patenting over claims 1-6 of U.S. Patent 6,689,262B2. The Examiner points out that the open-ended transitional term "comprising" can include Divisek's separator. Claim 1 as amended now reads "consisting of" a closed-ended term that does not allow the inclusion of Divisek's separator. Applicant submits herewith a terminal disclaimer which obviates this rejection

The claims being amended to more distinctly claim the invention, Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Respectfully submitted, Applicant James A. Senkiw, by his Attorney,

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Kathleen R. Terry Reg. No. 31884 (651) 659-9819 Krterry@visi.com

Please direct all correspondence to: Kathleen R. Terry 1666 Coffman Street Falcon Heights, MN 55108

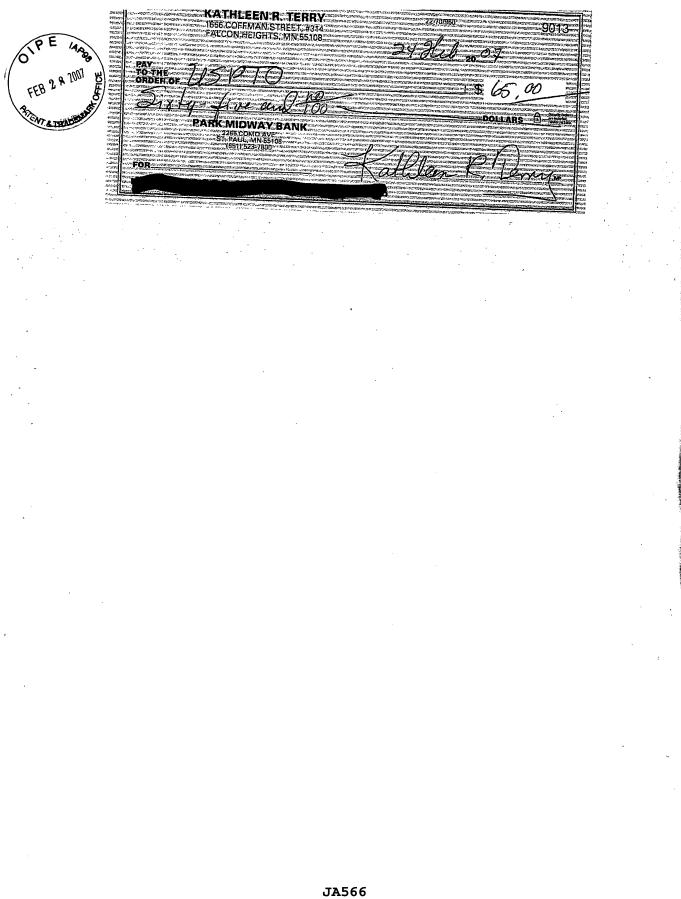
OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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FEB 2 8 2007 PTO/SB/25 (09-06) Approved for use through 03/31/2007. OMB 0651-0031 U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE ART COMBEN Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number. TERMINAL DISCLAIMER TO OBVIATE A PROVISIONAL DOUBLE PATENTING Docket Number (Optional) **REJECTION OVER A PENDING "REFERENCE" APPLICATION** In re Application of: James A. Senkiw Application No.: 10/732,326 Filed: 12/10/2003 For: FLOW THROUGH OXYGENATOR The owner\*, <u>AQUAINNOVATIONS, INC</u>, of <u>100</u> percent interest in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of any patent granted on pending reference Application Number <u>10/372,017</u>, filed on 02/21/2003 \_\_\_\_\_, as such term is defined in 35 U.S.C. 154 and 173, and as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed prior to the grant of any patent on the pending reference application. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and any patent granted on the reference application are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns. In making the above disclaimer, the owner does not disclaim the terminal part of any patent granted on the instant application that would extend to the expiration date of the full statutory term as defined in 35 U.S.C. 154 and 173 of any patent granted on said reference application, "as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed prior to the grant of any patent on the pending reference application," in the event that: any such patent: granted on the pending reference application: expires for failure to pay a maintenance fee, is held unenforceable, is found invalid by a court of competent jurisdiction, is statutorily disclaimed paper. in whole or terminally disclaimed under 37 CFR 1.321, has all claims canceled by a reexamination certificate, is reissued, or is in any manner terminated prior to the expiration of its full statutory term as shortened by any terminal disclaimer filed prior to its grant. Check either box 1 or 2 below, if appropriate. 1. For submissions on behalf of a business/organization (e.g., corporation, partnership, university, government agency, etc.), the undersigned is empowered to act on behalf of the business/organization. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section, 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon. 2. L The undersigned is an attorney or agent of record. Reg. No. 31,884 February 24, 2007 Signature Date Kathleen R. Jerry Typed-or printed name 651 659 9819 Telephone Number . 7. Terminal disclaimer fee under 37 CFR 1.20(d) is included. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038. \*Statement under 37 CFR 3.73(b) is required if terminal disclaimer is signed by the assignee (owner). Form PTO/SB/96 may be used for making this statement. See MPEP § 324. This collection of information is required by 37 CFR 1.321. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2. 02/28/2007 SSESHE1 00000035 10732326 65.00 OP ·. 01 FC:2814

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 568 of 1320



## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 569 of 1320

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and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

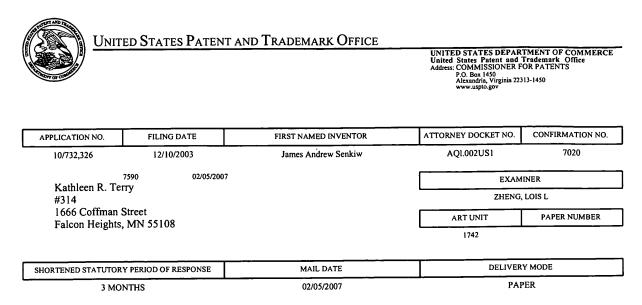
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## Please find below and/or attached an Office communication concerning this application or proceeding.

If NO period for reply is specified above, the maximum statutory period will apply and will expire 6 MONTHS from the mailing date of this communication.

PTOL-90A (Rev. 10/06)

	Application No.	Applicant(s)
Office Action Summary	10/732,326	SENKIW, JAMES ANDREW
	Examiner	Art Unit
	Lois Zheng	1742
The MAILING DATE of this communication a eriod for Reply	ppears on the cover sheet v	with the correspondence address
A SHORTENED STATUTORY PERIOD FOR REP WHICHEVER IS LONGER, FROM THE MAILING I - Extensions of time may be available under the provisions of 37 CFR 1 after SIX (6) MONTHS from the mailing date of this communication. - If NO period for reply is specified above, the maximum statutory perio - Failure to reply within the set or extended period for reply will, by statt Any reply received by the Office later than three months after the mail earned patent term adjustment. See 37 CFR 1.704(b).	DATE OF THIS COMMUN 1.136(a). In no event, however, may a d will apply and will expire SIX (6) MC ute, cause the application to become A	ICATION. a reply be timely filed INTHS from the mailing date of this communication. ABANDONED (35 U.S.C. § 133).
tatus		
1) Responsive to communication(s) filed on 09	November 2006	
	his action is non-final.	
3) Since this application is in condition for allow		tters, prosecution as to the merits is
closed in accordance with the practice under	Ex parte Quayle, 1935 C.	D. 11, 453 O.G. 213.
isposition of Claims		
4) Claim(s) <u>1-4 and 9</u> is/are pending in the appl	lication.	
4a) Of the above claim(s) is/are withdr		
5) Claim(s) is/are allowed.		
6) Claim(s) <u>1-4 and 9</u> is/are rejected.		
7) Claim(s) is/are objected to.		
8) Claim(s) are subject to restriction and	/or election requirement.	
pplication Papers		
9) The specification is objected to by the Examir	ner.	
10) The drawing(s) filed on is/are: a) ad		b by the Examiner.
Applicant may not request that any objection to th	ne drawing(s) be held in abeya	ance. See 37 CFR 1.85(a).
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11) The oath or declaration is objected to by the I	Examiner. Note the attache	ed Office Action or form PTO-152.
riority under 35 U.S.C. § 119		
12) Acknowledgment is made of a claim for foreig	gn priority under 35 U.S.C.	§ 119(a)-(d) or (f).
a) All b) Some * c) None of:		
1. Certified copies of the priority docume	nts have been received.	
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PTOL-326 (Rev. 08-06)

Office Action Summary

Part of Paper No./Mail Date 20070201

JA570

Page 2

### DETAILED ACTION

#### Status of Claims

1. Claims 1-2 are amended in view of the amendments filed 9 November 2006.

New claim 9 is added. Claims 5-8 remain withdrawn. Therefore, claims 1-4 and 9 are currently under examination.

#### Specification/Abstract

2. The amendment to the specification filed 9 November 2006 is not proper.

The amendment cannot be used to replace the abstract as requested by the applicant since the amendment includes continuing data which should be placed at the beginning of the specification.

### Claim Rejections - 35 USC § 112

3. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

4. Claims 1-4 and 9 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

In claim 1, the amended feature of "comprising an anode separated at a critical distance from a cathode <u>within an aqueous medium</u>" is vague and indefinite since it is unclear whether this limitation is directed to a cathode within an aqueous medium or to both an anode and a cathode each within an aqueous medium or to both an anode and a cathode each within an aqueous medium or to both an anode and an cathode within the same aqueous medium.

Page 3

In this office action, the examiner is interpreting this amended feature to mean that an anode is separated at a critical distance from a cathode and the cathode is within an aqueous medium or that both the anode and the cathode are each within an aqueous medium.

Claims 2-4 and 9 are also rejected since they depend on rejected claim 1.

## Claim Rejections - 35 USC § 102

5. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

6. Claims 1- 3 are rejected under 35 U.S.C. 102(b) as being anticipated by Divisek

et al. US 4,225,401(Divisek).

Divisek teaches a water electrolyzer for generating hydrogen and oxygen(abstract). The water electrolyzer comprises and anode separated at a distance from a cathode(Fig. 1). Divisek further teaches that the distance between the electrodes is about 1-3 mm(col. 3 lines 54-61).

Regarding instant claims 1 and 3, since the water electrolyzer of Divisek produces oxygen, the claimed oxygen microbubbles is inherently electrolytically generated when Divisek's water electrolyzer is in operation. In addition, Divisek teaches the claimed anode and cathode separated about 1-3 mm apart from each other, which reads on the claimed critical distance as recited in instant claim 3. The claimed power source is also inherently present in the water electrolyzer of Divisek.

JA572

Page 4

Fig. 1 of Divisek further shows that the water electrolyzer is placed within a conduit for flowing water Therefore, the water electrolyzer of Divisek meets the structural limitation of the instant claims 1 and 3. The examiner concludes that the electrolyzer of Divisek reads on the claimed flow-through oxygenator and the claimed emitter based on the broadest reasonable interpretation.

Regarding the amended feature in claim 1, the oxygen microbubbles are electrolytically generated from an aqueous medium in the water electrolyzer of Divisek. In addition, the cathode in the apparatus of Divisek is located within an aqueous medium as claimed.

Regarding claim 2, Divisek further teaches that the anode and the cathode are made of nickel(col. 4 lines 37-39), which meets the limitation of claimed metal anode and metal cathode.

Therefore, Divisek anticipates instant claims 1-3.

#### Claim Rejections - 35 USC § 103

7. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

8. Claim 4 is rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek.

The teachings of Divisek are discussed in paragraph 6 above.

Regarding instant claim 4, the distance of 1-3mm between the electrodes as

taught by Divisek encompasses the claimed critical distance of 0.045 to 0.060 inches.

JA573

Page 5

Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed critical distance from the disclosed range of Divisek would have been obvious to one skilled in the art since Divisek teaches the same utilities in its' disclosed critical distance range.

9. Claim 9 is rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek in view of Cairns et al. US 4,587,001(Cairns).

The teachings of Divisek are discussed in paragraph 6 above.

However, Divisek does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

Cairns teaches an cathode for use in an electrolytic cell(abstract). Cairns further teaches an titanium anode having a electro-catalytically active coating material comprising one or more oxides of platinum group metals such as platinum and iridium(col. 5 lines 15-25).

Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the anode of Cairns into the electrolyzer of Divisek as the anode since Cairns teaches that platinum group metal oxides is a good electro-catalytically active material for an anode of an electrolytic cell and the application of such coating on an anode is well known in the art(col. 5 lines 15-16 and 32-33).

#### **Double Patenting**

10. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct

from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., In re Berg, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); In re Goodman, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); In re Longi, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); In re Van Ornum, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); In re Vogel, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and In re Thorington, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

11. Claims 1-4 and 9 are rejected on the ground of nonstatutory obviousness-type

double patenting as being unpatentable over claims 1-6 of U.S. Patent No. 6,689,262

B2. Although the conflicting claims are not identical, they are not patentably distinct

from each other because the emitter of U.S. Patent No. 6,689,262 B2 is structurally the

same as the emitter of the claimed flow-through oxygenator. Even though U.S. Patent

No. 6,689,262 B2 does not explicitly teach the claimed flow through oxygenator, one of

ordinary skill in the art would have found it obvious to use the instant emitter in an

oxygenator as claimed since the emitter produces oxygen.

#### **Response to Arguments**

12. Applicant's arguments filed 9 November 2006 have been fully considered and are not persuasive.

In the remarks, applicant argues that oxygen microbubbles would not be formed, or if formed, would not persist, since the water electrolyzer of Divisek is operated at 300 to 600°C.

JA575

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 6

Page 7

The examiner does not find applicant's argument persuasive since applicant's argument is not backed with evidence data demonstrating that the water electrolyzer of Divisek is not capable of producing oxygen microbubbles as claimed. Therefore, applicant's argument is merely considered as conclusive statement. In addition, the operating temperature of the claimed apparatus is directed to the manner in which the claimed apparatus is operated, wherefore does not lend patentability to the instant apparatus of Divisek. Furthermore, the examiner asserts that the water electrolyzer of Divisek is inherently capable of be operated at ambient temperature, which is the desirable operating temperature of the claimed apparatus.

Applicant further argues that the electrodes of the instant invention are not separated by a separator and are within an aqueous medium.

The examiner does not find applicant's argument persuasive since claim 1 is vague and indefinite for the same reasons as stated in paragraph 4 above. In addition, claim 1 uses open-ended transitional phrase "comprising" which allows the presence of additional structural elements such as the separator as taught by Divisek.

#### **Terminal Disclaimer**

13. In the remarks filed 9 November 2006, applicant alleges that a terminal disclaimer was filed to overcome the non-statutory obviousness-type double patenting rejection. However, the examiner does not find this terminal disclaimer on the record. Therefore, the non-statutory obviousness-type double patenting rejection is maintained until proper terminal disclaimer is filed.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 8

## Conclusion

14. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Lois Zheng whose telephone number is (571) 272-1248. The examiner can normally be reached on 8:30am - 5:00pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Roy King can be reached on (571) 272-1244. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 9

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

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		FORM	First Named Inventor	James Andre	ew Senkiw	
			Art Unit	1742	·	
			Examiner Name	Lois L. Zhen	g	
	(to be used for	all correspondence after initial filing)	Attorney Docket Number	AQI.002US1		
	Total Number of	f Pages in This Submission 6		AQI.002051		
		El	NCLOSURES (Check all	l that apply)		
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	Signature	Killen K	Jane			
	Printed name	parmin L				-
		Kathleen R. Terry	<u> </u>			
	Date	November 6, 2006		Reg. No.	31,884	
	I hereby certify th sufficient postage the date shown b Signature	hat this correspondence is being fate as first class mail in an envelope	FICATE OF TRANSMISS	O or deposite	ed with the Ur	nited States Postal Service w Alexandria, VA 22313-1450
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					Date	November 6, 2006

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to hile (and by the USP10 to process) an application. Confidentially is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USP10. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 581



## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.10/732,326ApplicantJames A. Senkiw.Filed12/10/2003Art Unit1742ExaminerLois L. Zheng

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

## **RESPONSE AND AMENDMENT AMENDED FOR THE SECOND TIME**

Dear Ms. Zheng:

Enclosed please find a response, amended for the second time, to the Office action of 11/29/2005, with a complete listing of claims presented in proper ascending order and with the proper status identifiers.

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 4 of this paper.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA580

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# Amendments to the Specification:

Please replace the ABSTRACT of this application with the following:

This application is a continuation-in-part of United States Patent Application Number 10/372,017, filed on February 21, 2003, now United States Patent Number 6,689,262, issued February 10, 2004, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

**JA581** 

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

. . . <u>.</u>

#### Amendments to the Claims:

This listing of the claims will replace all prior versions, and listings, of claims in the application.

#### Listing of Claims

Claim 1. (Currently amended) A flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen <u>from an aqueous medium</u>, comprising an anode separated at a critical distance from a cathode <u>within an aqueous medium</u>, and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. (Currently amended) The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. (Original) The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 4. (Original) The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 5. (Withdrawn) The product of claim 1 wherein the water is upersaturated with oxygen and of an approximately neutral pH.

Claim 6. (Withdrawn) A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

Claim 7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

Claim 8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

Claim 9. (New) The emitter of claim 1 wherein the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### **REMARKS/ARGUMENTS**

The Examiner has rejected pending claims 1 and 3 under U.S.C. § 102(b) as being anticipated by Divisek et al. US 4,225,401 ("Divisek"). Applicant respectfully disagrees.

In order to anticipate a claim, a reference must include each and every element or its equivalent, either explicitly or inherently. The claims as filed comprise the elements of "microbubbles," which would not be formed at the temperatures recited by Divisek, that is, 300° to 600° C (Divisek, column 3, line9), or if formed, would not persist. While not explicitly recited in the specification or claims, the present invention is designed to operate at ambient temperatures. (See for example, specification, Examples 5 and 6 on pages 11-15.)

Claim 1 has been amended to emphasize that unlike Divisek, the electrodes are not separated by a separator, but are within an aqueous medium. The substrate for electrolysis is emphasized to be an aqueous medium as well, to further distinguish the claims from Divisek. Divisek uses molten NaOH as an electrolyte with water vapor introduced as a substrate for electrolysis. (Divisek, column 4, line 56.) Applicant believes that claim 1 is now allowable.

The Examiner has rejected claim 4 under U.S.C. §103 (a) as being obvious from Divisek. Divisek is actually silent as to the distance between electrodes. On column 3, lines 57-61, Divisek states that "distance between the electrodes which merely corresponds to the thickness of the separator are possible, in other words, for all practical purposes, this distance may amount to about 1-3 mm." While 1-3 mm overlaps with the critical distance recited in claim 4, claim 4 depends on claim 1 and includes all the elements of claim 1, since claim1 has now been distinguished from Divisek, it is submitted that the rejection is of claim 4 now longer applies.

The Examiner has rejected claim 2 under 35 U.S.C. § 103 (a) as being obvious from Divisek in view of Cairns et al U.S. 4,587,001. Claim 2 being dependant from claim 1, it should be read with all the elements of claim 1. New claim 5 has been added to delete the elements "platinum and iridium" from the claim 2 and present them in a new dependant claim. The invention to be operative is not dependant on any specific anodes and cathodes (specification, page 4, line 1-8) but the platinum and iridium electrodes are more durable and thence comprise the best mode of making the invention. Applicant believes that claim 2 and new claim 5 are now allowable.

The Examiner has rejected claim 1-4 on the ground of non-statutory obviousness-type double patenting over claims 1-6 of U.S. Patent 6,689,262B2. Applicant submits herewith a terminal disclaimer which obviates this rejection.

4

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

The claims being amended to more distinctly claim the invention and listing of the withdrawn claims added, Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Respectfully submitted, Applicant James A. Senkiw, by his Attorney,

Kathleen R. Terry Reg. No. 31884 (651) 659-9819 Krterry@visi.com

Please direct all correspondence to: Kathleen R. Terry 1666 Coffman St. #314 Falcon Heights, MN 55108

**JA584** 

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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	ATENT APPL	Substitute for			RECORD		r Docket Number 32,326		ing Date 10/2003	To be Mail
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process) an application. Confidentiality is governed by 35 0.5.C. 122 and 37 CFN 1.14. This collection is estimated to take 12 minutes to complete, including gathering preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.** If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2

JA585

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 588 of 1320

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			UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F P.O. Box 1450 Alexandria, Virginia 223 www.uspto.gov	Trademark Office OR PATENTS
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326	12/10/2003	James Andrew Senkiw	AQ1.002US1	7020
. 75	90 10/13/2006		EXAM	INER
Kathleen R. Te #314	erry		ZHENG,	LOIS L
1666 Coffman S	Street		ART UNIT	PAPER NUMBER
Falcon Heights,	MN 55108		1742	
			DATE MAILED: 10/13/200	6

Please find below and/or attached an Office communication concerning this application or proceeding.

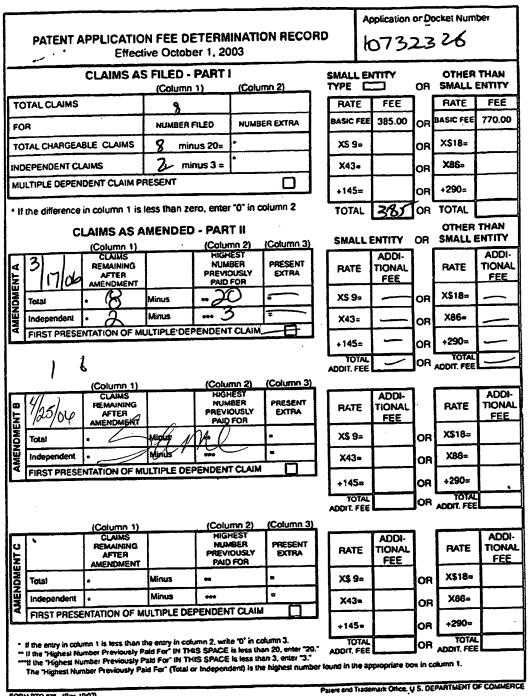
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	Application No.	Applicant(s)	
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<i>Notice of Non-Compliant Amendment (37 CFR 1.121)</i>	Examiner	Art Unit	
The MAILING DATE of this, communication a	pears on the cover sheet wit	h the correspondence	address
e amendment document filed on <u>125</u> 004 is considered FR 1.121. In order for the amendment document to b	non-compliant because it h c compliant, correction of th	as failed to meet the r le following item(s) is r	requirements of 37 required.
HE FOLLOWING MARKED (X) ITEM(S) CAUSE THI 1. Amendments to the specification: A. Amended paragraph(s) do not includ B. New paragraph(s) should not be und C. Other	le markings.	IT TO BE NON-COM	PLIANT:
<ul> <li>2. Abstract:</li> <li>A. Not presented on a separate sheet.</li> <li>B. Other</li> </ul>	37 CFR 1.72.		
<ul> <li>3. Amendments to the drawings:</li> <li>A. The drawings are not properly identi "Annotated Sheet" as required by 37</li> <li>B. The practice of submitting proposed showing amended figures, without n</li> <li>C. Other</li> </ul>	CFR 1.121(d). drawing correction has been	n eliminated. Replace	ment drawings
<ul> <li>4. Amendments to the claims:</li> <li>A. A complete listing of all of the claims</li> <li>B. The listing of claims does not include</li> <li>C. Each claim has not been provided w of each claim cannot be identified. I number by using one of the following (Previously presented), (New), (Not</li> <li>D. The claims of this amendment pape</li> <li>E. Other: <u>ILUL</u> y presented is a set of the set of</li></ul>	e the text of all pending clain ith the proper status identific Note: the status of every cla g status identifiers: (Original entered), (Withdrawn) and (	er, and as such, the in aim must be indicated ), (Currently amended Withdrawn-currently a	dividual status after its claim ), (Canceled), mended).
r further explanation of the amendment format requ p://www.uspto.gov/web/offices/pac/dapp/opla/preog	ired by 37 CFR 1.121, see M		
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Applicant is given one month, or thirty (30) days, corrected section of the non-compliant amendment amendment is one of the following: a preliminary a request for continued examination (RCE) under 3 period under 37 CFR 1.103(a) or (c), and an amendment	whichever is longer, from th ent in compliance with 37 Cl amendment, a non-final ame 7 CFR 1.114), a supplement	e mail date of this not FR 1.121, if the non-co endment (including a s al amendment filed w	ice to supply the ompliant submission for a
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FORM PTO-875 (Bey 10:03)

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 591 of 1320

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· · · ·		Application Number	10/732.326
	CHANGE OF CORRESPONDENCE ADDRESS	Filing Date	12/10/2003
	Application	First Named Inventor	James Andrew Senkiw
	Address to:	Art Unit	1742
	Commissioner for Patents P.O. Box 1450	Examiner Name	Lois L. Zheng
	Alexandria, VA 22313-1450	Attorney Docket Number	AQI.002US1
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	This form cannot be used to change the data associated data associated with an existing Customer Number use I am the: Applicant/Inventor Assignee of record of the entire interest. Statement under 37 CFR 3.73(b) is encl	e "Request for Customer Nun	To change the nber Data Change" (PTO/SB/124).
	Attorney or agent of record. Registration	Number <u>31,884</u>	
	Registered practitioner named in the app executed oath or declaration. See 37 CF	plication transmittal letter in a FR 1.33(a)(1). Registration Nu	n application without an umber
	Signature Autoen R. Lenne Typed or Printed Name Kathleen R. Terrry	(	
		Telephone 651 659 9819	
	Date 24 July 2006 NOTE: Signatures of all the inventors or assignees of record of the entire inte forms if more than one signature is required, see below*.		uired. Submit multiple
	Total of F forms are submitted.		
	This collection of information is required by 37 CFR 1.33. The information is to process) an application. Confidentiality is governed by 35 U.S.C. 122 and	required to obtain or retain a benefit	by the public which is to file (and by the USPTO tion is estimated to take 3 minutes to complete.

This collection of information is required by 37 CFR 1.33. The information is required to obtain or retain a benefit by the public which is to the (and by the USP10 to process) an application. Confidentiality is gowened by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USP10. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 592 of 1320

TRANSMITTAL FORM         sused for all correspondence after initia         number of Pages in This Submission         iee Transmittal Form         Fee Attached         wmendment/Reply         After Final         After Final         After Submission of Time Request         Express Abandonment Request         formation Disclosure Statement         Certified Copy of Priority	First Named Inventor     James A. Senkiw       Art Unit     1742       Examiner Name     Lois L. Zhebo
Fee Transmittal Form Fee Attached After Final Affidavits/declaration(s) Extension of Time Request Express Abandonment Request formation Disclosure Statement Certified Copy of Priority	□       Drawing(s)       □       After Allowance Communication to TC         □       Licensing-related Papers       □       Appeal Communication to Board of Appeals and Interferences         □       Petition       □       Appeal Communication to TC (Appeal Notice, Brief, Repty Brief)         □       Petition to Convert to a Provisional Application       □       Proprietary Information         □       Power of Attorney, Revocation Change of Correspondence Address       □       Status Letter         □       Terminal Disclaimer       ✓       Other Enclosure(s) (please Identify below):         □       CD, Number of CD(s)       □
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Document(s) Reply to Missing Parts/ Incomplete Application Reply to Missing Parts under 37 CFR 1.52 or 1.53	Remarks Amended response with Notice of Non-Compliant Amendment
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Kathfeen R. Terry 19 April 2006	Reg. No. 31,884
certify that this correspondence is	ERTIFICATE OF TRANSMISSION/MAILING being facsimile transmitted to the USPTO or deposited with the United States Postal Service with nvelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on
	e Kathleen R. Terry 19 April 2006

This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentially is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.10/732,326ApplicantJames A. Senkiw.Filed12/10/2003Art Unit1742ExaminerLois L. Zheng

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

### AMENDED RESPONSE AND AMENDMENT

Dear Ms. Zheng:

Enclosed please find an amended response to the Office action of 11/29/2005, with the claims presented in proper ascending order:

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 4 of this paper.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

1

## Amendments to the Specification:

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This application is a continuation-in-part of United States Patent Application Number 10/372,017, filed on February 21, 2003, now United States Patent Number 6,689,262, issued February 10, 2004, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

#### Amendments to the Claims:

This listing of the claims will replace all prior versions, and listings, of claims in the application.

#### **Listing of Claims**

Claim 1. (Currently amended) A flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen <u>from an aqueous medium</u>, comprising an anode separated at a critical distance from a cathode <u>within an aqueous medium</u>, and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. (Currently amended) The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. (Original) The critical distance of claim 1 which is 0.005 to 0.140 inches.

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Claim 7. (Withdrawn) The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

Claim 8. (Withdrawn) A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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#### **REMARKS/ARGUMENTS**

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In order to anticipate a claim, a reference must include each and every element or its equivalent, either explicitly or inherently. The claims as filed comprise the elements of "microbubbles," which would not be formed at the temperatures recited by Divisek, that is, 300° to 600° C (Divisek, column 3, line9), or if formed, would not persist. While not explicitly recited in the specification or claims, the present invention is designed to operate at ambient temperatures. (See for example, specification, Examples 5 and 6 on pages 11-15.)

Claim 1 has been amended to emphasize that unlike Divisek, the electrodes are not separated by a separator, but are within an aqueous medium. The substrate for electrolysis is emphasized to be an aqueous medium as well, to further distinguish the claims from Divisek. Divisek uses molten NaOH as an electrolyte with water vapor introduced as a substrate for electrolysis. (Divisek, column 4, line 56.) Applicant believes that claim 1 is now allowable.

The Examiner has rejected claim 4 under U.S.C. §103 (a) as being obvious from Divisek. Divisek is actually silent as to the distance between electrodes. On column 3, lines 57-61, Divisek states that "distance between the electrodes which merely corresponds to the thickness of the separator are possible, in other words, for all practical purposes, this distance may amount to about 1-3 mm." While 1-3 mm overlaps with the critical distance recited in claim 4, claim 4 depends on claim 1 and includes all the elements of claim 1, since claim1 has now been distinguished from Divisek, it is submitted that the rejection is of claim 4 now longer applies.

The Examiner has rejected claim 2 under 35 U.S.C. § 103 (a) as being obvious from Divisek in view of Cairns et al U.S. 4,587,001. Claim 2 being dependant from claim 1, it should be read with all the elements of claim 1. New claim 5 has been added to delete the elements "platinum and iridium" from the claim 2 and present them in a new dependant claim. The invention to be operative is not dependant on any specific anodes and cathodes (specification, page 4, line 1-8) but the platinum and iridium electrodes are more durable and thence comprise the best mode of making the invention. Applicant believes that claim 2 and new claim 5 are now allowable.

The Examiner has rejected claim 1-4 on the ground of non-statutory obviousness-type double patenting over claims 1-6 of U.S. Patent 6,689,262B2. Applicant submits herewith a terminal disclaimer which obviates this rejection.

4

The claims being amended to more distinctly claim the invention and listing of the withdrawn claims added, Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Respectfully submitted, Applicant James A. Senkiw, by his Attorney,

Hleen

Kathleen R. Terry Reg. No. 31884 (651) 659-9819 Krterry@visi.com

Please direct all correspondence to: Kathleen R. Terry 2417 Como Avenue St. Paul, MN 55108-1459

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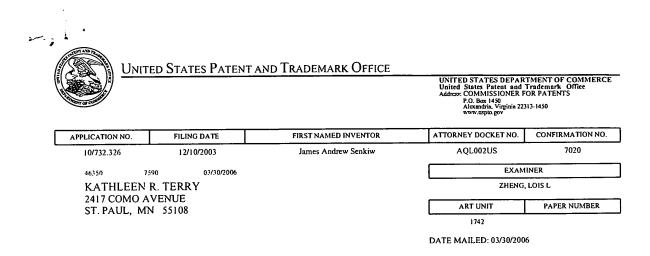
OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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<ul> <li>B. The practice of submitting proposed showing amended figures, without m</li> <li>C. Other</li> </ul>	drawing correction has been arkings in compliance with	n eliminated. Replacement c	drawings		
C. Other	incompliance with	137 CFR 1.84 are required.	-		
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<ul> <li>B. The listing of claims does not include</li> <li>C. Each claim has not been provided wi of each claim cannot be identified.</li> </ul>	the text of all pending clair ith the proper status identifi	ns (including withdrawn claim	is)		
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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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Please find below and/or attached an Office communication concerning this application or proceeding.

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PTO-90C (Rev. 10/03)

Notice of Non-Compliant	Application No.	Applicant(s)	Applicant(s)		
Amendment (37 CFR 1.121)	Examiner	Art Unit			
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<ul> <li>2. Abstract:</li> <li>A. Not presented on a separate sheet.</li> <li>B. Other</li> </ul>	37 CFR 1.72.				
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<ul> <li>4. Amendments to the claims:</li> <li>A. A complete listing of all of the claims</li> <li>B. The listing of claims does not include</li> <li>C. Each claim has not been provided w of each claim cannot be identified. N number by using one of the following (Previously presented), (New), (Not D. The claims of this amendment paper F. Other: <u>Claims</u> 6, 7, 8 or 5. The amendment is unsigned event size to</li> </ul>	e the text of all pending cl ith the proper status iden Note: the status of every status identifiers: (Origin entered), (Withdrawn) and have not been presented in Electory, Clarim	ifier, and as such, the individual sta claim must be indicated after its cla al), (Currently amended), (Cancele I (Withdrawn-currently amended). in ascending numerical order.	aim ed),		
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Extensions of time are available under 37 CFR amendment or an amendment filed in response to Failure to timely respond to this notice will resu Abandonment of the application if the non-co	1.136(a) <u>only</u> if the non-o o a <i>Quayle</i> action.	compliant amendment is a non-final			
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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

10/732,326 Applicant James A. Senkiw. 12/10/2003 Art Unit 1742 Lois L. Zheng Examiner

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

### **RESPONSE AND AMENDMENT**

Dear Ms. Zheng:

Enclosed please find a Request for Extension of Time. In response to the Office action of 11/29/2005, please amend the above-identified application as follows:

Amendments to the Specification begin on page 2 of this paper.

Amendments to the Claims are reflected in the listing of claims which begins on page 3 of this paper.

Remarks begin on page 4 of this paper.

**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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## Amendments to the Specification:

Please replace the ABSTRACT of this application with the following:

This application is a continuation-in-part of United States Patent Application Number 10/372,017, filed on February 21, 2003, now United States Patent Number 6,689,262, issued February 10, 2004, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### Amendments to the Claims:

This listing of the claims will replace all prior versions, and listings, of claims in the application.

### **Listing of Claims**

Claim 1. (Currently amended) A flow-through oxygen ator comprising an emitter for electrolytic generation of microbubbles of oxygen from an aqueous medium, comprising an anode separated at a critical distance from a cathode within an aqueous medium, and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. (Currently amended) The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. (Original) The critical distance of claim 1 which is 0.005 to 0.140 inches.

Claim 4. (Original) The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 5. (Newly presented) The emitter of claim 1 <u>wherein the anode is platinum</u> and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

#### **REMARKS/ARGUMENTS**

The Examiner has rejected pending claims 1 and 3 under U.S.C. § 102(b) as being anticipated by Divisek et al. US 4,225,401 ("Divisek"). Applicant respectfully disagrees.

In order to anticipate a claim, a reference must include each and every element or its equivalent, either explicitly or inherently. The claims as filed comprise the elements of "microbubbles," which would not be formed at the temperatures recited by Divisek, that is, 300° to 600° C (Divisek, column 3, line9), or if formed, would not persist. While not explicitly recited in the specification or claims, the present invention is designed to operate at ambient temperatures. (See for example, specification, Examples 5 and 6 on pages 11-15.)

Claim 1 has been amended to emphasize that unlike Divisek, the electrodes are not separated by a separator, but are within an aqueous medium. The substrate for electrolysis is emphasized to be an aqueous medium as well, to further distinguish the claims from Divisek. Divisek uses molten NaOH as an electrolyte with water vapor introduced as a substrate for electrolysis. (Divisek, column 4, line 56.) Applicant believes that claim 1 is now allowable.

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The Examiner has rejected claim 2 under 35 U.S.C. § 103 (a) as being obvious from Divisek in view of Cairns et al U.S. 4,587,001. Claim 2 being dependant from claim 1, it should be read with all the elements of claim 1. New claim 5 has been added to delete the elements "platinum and iridium" from the claim 2 and present them in a new dependant claim. The invention to be operative is not dependant on any specific anodes and cathodes (specification, page 4, line 1-8) but the platinum and iridium electrodes are more durable and thence comprise the best mode of making the invention. Applicant believes that claim 2 and new claim 5 are now allowable.

The Examiner has rejected claim 1-4 on the ground of non-statutory obviousness-type double patenting over claims 1-6 of U.S. Patent 6,689,262B2. Applicant submits herewith a terminal disclaimer which obviates this rejection.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 605 of 1320

Appl. No. 10/732,326 Amendment dated March 15, 2006 Response to Office Action of November 29, 2005

The claims being amended to more distinctly claim the invention, Applicant respectfully requests that a timely Notice of Allowance be issued in this case.

Respectfully submitted, Applicant James A. Senkiw, by his Attorney,

Kathleen R. Terry Reg. No. 31884 (651) 659-9819 Krterry@visi.com

Please direct all correspondence to: Kathleen R. Terry 2417 Como Avenue St. Paul, MN 55108-1459

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CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 606 of 1320

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**OWT Ex. 2118** Tennant Company v. OWT IPR2021-00625

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CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 607 of 1320

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This collection of information is required by 37 CFR 1.5. The information is required to obtain or retain a benefit by the public which is to file (and by the USP10 to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to 2 hours to complete, including gathering, preparing, and submitting the complete application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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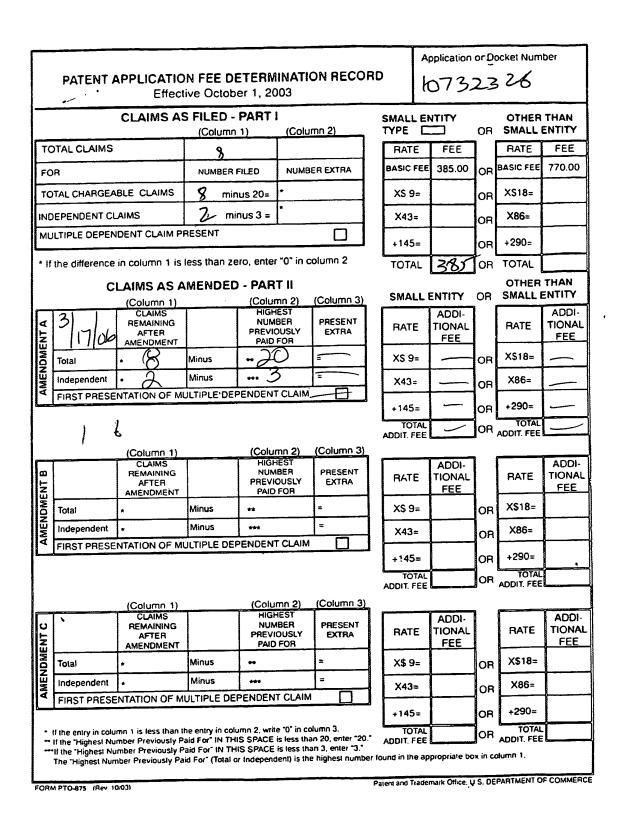
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Page 608

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 609 of 1320

KATHLEEN R. TERRY 2417 COMO AVENUE SAINT PAUL, MN 55108 22-70/960 8772 15 March 2006 PAV TO THE ORDER OF -00 5 he DOLLARS Mo 100 1100 SAINT ANTHONY PARK STATE BANK OMO AVE: L, MN 65108 623-7800 FOR 002451 Real States

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625



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APPLICATION NO. FILING DATE FI	ST NAMED INVENTOR	TTORNEY DOCKET NO.	CONFIRMATION NO.
10/732,326 12/10/2003	ames Andrew Senkiw	AQL002US	7020
46350 7590 11/29/2005	Г	EXAMIN	IER
KATHLEEN R. TERRY		ZHENG, L	OIS L
2417 COMO AVENUE ST. PAUL, MN 55108	Г	ARTUNIT	PAPER NUMBER

Please find below and/or attached an Office communication concerning this application or proceeding.

PTO-90C (Rev. 10/03)

JA609

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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	Application No.	Applicant(s)
Office Action Summary	10/732,326	SENKIW, JAMES ANDREW
	Examiner	Art Unit
	Lois Zheng	1742
The MAILING DATE of this communication a Period for Reply	ppears on the cover she	et with the correspondence address
A SHORTENED STATUTORY PERIOD FOR REF WHICHEVER IS LONGER, FROM THE MAILING - Extensions of time may be available under the provisions of 37 CFR after SIX (6) MONTHS from the mailing date of this communication. - If NO period for reply is specified above, the maximum statutory perio - Failure to reply within the set or extended period for reply will, by stat Any reply received by the Office later than three months after the ma earned patent term adjustment. See 37 CFR 1.704(b).	DATE OF THIS COMM 1.136(a). In no event, however, n od will apply and will expire SIX (6 ute, cause the application to beco	UNICATION. hay a reply be timely filed ) MONTHS from the mailing date of this communication. me ABANDONED (35 U.S.C. § 133).
Status		
1) $\boxtimes$ Responsive to communication(s) filed on <u>10</u>	December 2003.	
	nis action is non-final.	
3) Since this application is in condition for allow closed in accordance with the practice unde	vance except for formal	
Disposition of Claims		,
4) Claim(s) <u>1-8</u> is/are pending in the application	٦.	
4a) Of the above claim(s) <u>5-8</u> is/are withdraw		
5) Claim(s) is/are allowed.	r.	
6) Claim(s) <u>1-4</u> is/are rejected.		
7) Claim(s) is/are objected to.	۰.	
8) Claim(s) are subject to restriction and	l/or election requiremen	t.
Application Papers		
9) The specification is objected to by the Exami		
10) The drawing(s) filed on is/are: a) a		
Applicant may not request that any objection to the		
Replacement drawing sheet(s) including the correct	•	
11) The oath or declaration is objected to by the	Examiner. Note the atta	iched Office Action or form PTO-152.
Priority under 35 U.S.C. § 119		
12) Acknowledgment is made of a claim for forei	gn priority under 35 U.S	.C. § 119(a)-(d) or (f).
a) All b) Some * c) None of:		
1. Certified copies of the priority docume		
2. Certified copies of the priority docume		
3. Copies of the certified copies of the practice application from the International Bure		been received in this National Stage
* See the attached detailed Office action for a li		not received
Attachment(s)	_	
<ol> <li>Notice of References Cited (PTO-892)</li> <li>Notice of Draftsperson's Patent Drawing Review (PTO-948)</li> </ol>		view Summary (PTO-413) r No(s)/Mail Date
<ul> <li>A Nonce of Dransperson's Patent Drawing Review (P10-948)</li> <li>A Information Disclosure Statement(s) (PTO-1449 or PTO/SB/C Paper No(s)/Mail Date <u>19 July 2004</u>.</li> </ul>		e of Informal Patent Application (PTO-152)
S. Patent and Trademark Office TOL-326 (Rev. 7-05) Office	Action Summary	Part of Paper No./Mail Date 11212005

**JA610** 

Part of Paper No./Mail Date 11212005

Page 2

## DETAILED ACTION

#### Election/Restrictions

- 1. Restriction to one of the following inventions is required under 35 U.S.C. 121:
  - I. Claims 1-4, drawn to a flow-through oxygenator, classified in class 204, subclass 242.
  - II. Claim 5, drawn to an oxygen supersaturated water product, classified in class 205, subclass 633.
  - III. Claims 6-7, drawn to a method for enhancing growth of plants, classified in class 47, subclass 58.1 SC.
  - IV. Claim 8, drawn to a method for treating waste water, classified in class
     205, subclass 742.

2. Inventions I and II are related as apparatus and product made. The inventions in this relationship are distinct if either or both of the following can be shown: (1) that the apparatus as claimed is not an obvious apparatus for making the product and the apparatus can be used for making a different product or (2) that the product as claimed can be made by another and materially different apparatus (MPEP § 806.05(g)). In this case the oxygen supersaturated water can be made by another and materially different apparatus apparatus such as an non-electrochemical fluid aeration device.

3. Inventions III and I are unrelated. Inventions are unrelated if it can be shown that they are not disclosed as capable of use together and they have different modes of operation, different functions, or different effects (MPEP § 806.04, MPEP § 808.01). In the instant case the different inventions have different function. Invention I is drawn to

**JA611** 

Page 3

an oxygenator apparatus while Invention III is drawn to a process for enhancing growth of plants.

4. Inventions IV and I are related as process and apparatus for its practice. The inventions are distinct if it can be shown that either: (1) the process as claimed can be practiced by another materially different apparatus or by hand, or (2) the apparatus as claimed can be used to practice another and materially different process. (MPEP § 806.05(e)). In this case the apparatus of Invention I can be used to practice another and materially difference process, such as a process to produce oxygen.

5. Because these inventions are distinct for the reasons given above and have acquired a separate status in the art because of their recognized divergent subject matter, restriction for examination purposes as indicated is proper.

6. During a telephone conversation with Kathleen R. Terry on 15 November 2005 a provisional election was made without traverse to prosecute the invention of group I, claims 1-4. Affirmation of this election must be made by applicant in replying to this Office action. Claims 58 are withdrawn from further consideration by the examiner, 37 CFR 1.142(b), as being drawn to a non-elected invention.

#### Claim Rejections - 35 USC § 102

7. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 4

8. Claims 1 and 3 are rejected under 35 U.S.C. 102(b) as being anticipated by Divisek et al. US 4,225,401(Divisek).

Divisek teaches a water electrolyzer for generating hydrogen and oxygen(abstract). The water electrolyzer comprises and anode separated at a distance from a cathode(Fig. 1). Divisek further teaches that the distance between the electrodes is about 1-3 mm(col. 3 lines 54-61).

Regarding instant claims 1 and 3, since the water electrolyzer of Divisek produces oxygen, the claimed oxygen microbubbles is inherently electrolytically generated when Divisek's water electrolyzer is in operation. In addition, Divisek teaches the claimed anode and cathode separated about 1-3 mm apart from each other, which reads on the claimed critical distance as recited in instant claim 3. The claimed power source is also inherently present in the water electrolyzer of Divisek. Fig. 1 of Divisek further shows that the water electrolyzer is placed within a conduit for flowing water Therefore, the water electrolyzer of Divisek meets the structural limitation of the instant claims 1 and 3. The examiner concludes that the electrolyzer of Divisek reads on the claimed flow-through oxygenator and the claimed emitter based on the broadest reasonable interpretation.

Therefore, Divisek anticipates instant claims 1 and 3.

#### Claim Rejections - 35 USC § 103

9. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

Page 5

invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

Claim 4 is rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek.
 The teachings of Divisek are discussed in paragraph 8 above.

Regarding instant claim 4, the distance of 1-3mm between the electrodes as taught by Divisek encompasses the claimed critical distance of 0.045 to 0.060 inches.

Therefore, a prima facie case of obviousness exists. See MPEP 2144.05. The selection of claimed critical distance from the disclosed range of Divisek would have been obvious to one skilled in the art since Divisek teaches the same utilities in its' disclosed critical distance range.

11. Claim 2 is rejected under 35 U.S.C. 103(a) as being unpatentable over Divisek in view of Cairns et al. US 4,587,001(Cairns).

The teachings of Divisek are discussed in paragraph 8 above. Divisek further teaches that the anode and the cathode are made of nickel(col. 4 lines 37-39).

However, Divisek does not explicitly teach the claimed anode being platinum and iridium oxide on a support.

Cairns teaches an cathode for use in an electrolytic cell(abstract). Cairns further teaches an titanium anode having a electro-catalytically active coating material comprising one or more oxides of platinum group metals such as platinum and iridium(col. 5 lines 15-25).

Therefore, it would have been obvious to one of ordinary skill in the art to have incorporated the anode of Cairns into the electrolyzer of Divisek as the anode since Cairns teaches that platinum group metal oxides is a good electro-catalytically active

**JA614** 

Page 6

material for an anode of an electrolytic cell and the application of such coating on an

anode is well known in the art(col. 5 lines 15-16 and 32-33).

#### Double Patenting

12. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., In re Berg, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); In re Goodman, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); In re Longi, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); In re Van Ornum, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); In re Vogel, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and In re Thorington, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

13. Claims 1-4 are rejected on the ground of nonstatutory obviousness-type double

patenting as being unpatentable over claims 1-6 of U.S. Patent No. 6,689,262 B2.

Although the conflicting claims are not identical, they are not patentably distinct from

each other because the emitter of U.S. Patent No. 6,689,262 B2 is structurally the same

as the emitter of the claimed flow-through oxygenator. Even though U.S. Patent No.

6,689,262 B2 does not explicitly teach the claimed flow through oxygenator, one of

ordinary skill in the art would have found it obvious to use the instant emitter in an

oxygenator as claimed since the emitter produces oxygen.

JA615

Page 7

### Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Lois Zheng whose telephone number is (571) 272-1248. The examiner can normally be reached on 8:30am - 5:00pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Roy King can be reached on (571) 272-1244. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

> ROY KING SUPERVISORY PATENT EXAMINER TECHNOLOGY CENTER 1700

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1P JUL 1 9 2004 PTO/SB08A (08-03) Approved for use through 07/31/2006. OMB 0651-0001 U.S. Patent and Trademark Office; U.S. DEPART MENT OF COMMERCE Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number. Inderes bstitute for form 1449/PTO Complete if Known Application Number 10/732,326 Filing Date December 10. 2003 INFORMATION DISCLOSURE First Named Inventor James Andrew Senkiw STATEMENT BY APPLICANT Art Unit 1742 (Use as many sheets as necessary) Examiner Name Sheet 1 of 2 Attorney Docket Number AQI.002US1 U. S. PATENT DOCUMENTS Examiner Initials\* Cite No.1 Document Number Publication Date MM-DD-YYYY Name of Patentee or Applicant of Cited Document Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear Number-Kind Code<sup>2 (# taxe</sup> <sup>US-</sup> 6,689,262 02-10-2004 Senkiw <sup>US-</sup> 6,394,429 01 05-78-2002 Gvan - Calua 11-13-2001 Zappi et al. <sup>US-</sup> 6,315,886B1 <sup>US-</sup> 5,982,609 11-09-1959 Evans <sup>US-</sup> 5,534,143 07-09-1986 Portier et al. <sup>US-</sup> 4,252,856 03-24-1981 5252 US-115 lis TIS. US. US-US-US. ÚŚ. US-US-US-US-FOREIGN PATENT DOCUMENTS Publication Na Date Applic Examiner Cite No. Foreign Patent Document Pages, Columns, Lines, Where Relevant Passages Or Relevant Figures Appear Name of Patentee or Applicant of Cited Document MM-DD-YYYY T<sup>4</sup> Country Code<sup>3</sup> Number<sup>4</sup> Kind Code<sup>5</sup> (# known) Examine Ħ Signature Date nf ~ "[2] Considered  $' \circ S$ 

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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. 1 Applicant's unique citation designation number (optional). 2 Applicant is to place a check mark here if English language Transistion is attached. This collection of Information is required by 37 CFR 1.99. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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*	Α	US-4,225,401	09-1980	Divisek et al.			205/354
*	В	US-4,587,001	05-1986	Cairns et al.			204/290.14
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U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

Notice of References Cited

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 624 of 1320

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
S1	204	(205/633-636).ccls.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/14 09:36 <sup>.</sup>
S2	3	S1 and microbubbles	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/14 09:37
S3	0	S1 and micro adj bubbles	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/14 09:37
S4	3	S1 and critical distance	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2005/11/14 09:38
S5	2	"6689262".pn.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/14 09:39
S6	10	("3975269"   "4012319"   "4732661"   "4908109"   "5049252"   "5182014"   "5534143"   "6315886"   "6394429"   "6471873"   "WO 9521795").PN.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 09:46
S7	7813	anode with cathode with distance	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 09:46
S8	140	S7 and water adj (electrolyz\$4 or electrolysis)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 09:47
S9	105	S7 and water adj (electrolyz\$4 or electrolysis) and oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 09:47
S11	76	S9 and distance with (inch or in or micrometer or micron or "mu.m" or millimeter or mm or centimeter or cm)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 10:07

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# CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 625 of 1320

S12	7813	anode with cathode with distance	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/14 12:46
S13	105	S12 and water adj (electrolyz\$4 or electrolysis) and oxygen	US-PGPUB; USPAT; USOCR	OR _	ON	2005/11/14 12:46
S14	76	S13 and distance with (inch or in or micrometer or micron or "mu. m" or millimeter or mm or centimeter or cm)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:53
S15	8	S14 and anode with (platnium or Pt or iridium or Ir) with oxide	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:27
S16	13	(growth or yield) with plants and (supersaturated or (super adj saturated)) adj water	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:29
S17	1	"5887383".pn.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/15 09:26
S18	1	"6004450".pn.	US-PGPUB; USPAT; USOCR	OR ·	ON	2005/11/15 09:27
S19	3230	oxygenator	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:34
S20	279	S19 and (oxygen or "O.sub.2") near5 (bubbles or microbubbles)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:35
S21	40	S20 and (electrode or anode or cathode)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:35
S23		S21 and distance with (inch or in or micrometer or micron or "mu. m" or millimeter or mm or centimeter or cm)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:34
S24	1253	oxygenator	EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 10:35
S25	0	S24 and (oxygen or "O.sub.2") near5 (bubbles or microbubbles)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:35
S26	0	S24 and (electrode or anode or cathode)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:35

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S27	204	(205/633-636).ccls.	US-PGPUB;	OR	ON	2005/11/21 10:49
			USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB			
S28	18	(205/633-636).ccls. and bubbles with oxygen	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 10:49
S29	7824	anode with cathode with distance	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 10:53
S30	105	S29 and water adj (electrolyz\$4 or electrolysis) and oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 14:07
S31	76	S30 and distance with (inch or in or micrometer or micron or "mu. m" or millimeter or mm or centimeter or cm)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 14:07
S32	6	("4252856" "5534143" "5982609"  "6315886" "6394429" "6689262" ).PN.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 11:13
S35	1	"4048047".pn.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 11:13
S37	860	(205/628-639).ccls.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 14:06
S38	350	S37 and water near3 (electrolyz\$4 or electrolysis) and oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 14:07
S39	40	S38 and distance with (inch or in or micrometer or micron or "mu. m" or millimeter or mm or centimeter or cm)	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:06
S40	10	("3975269"   "4012319"   "4732661"   "4908109"   "5049252"   "5182014"   "5534143"   "6315886"   "6394429"   "6471873"   "WO 9521795").PN.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 14:14
S42	187	supersaturated adj water	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:06

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**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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S43	19	supersaturated adj water with oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/22 09:53
S44	4	(growth or yield) with plants and (supersaturated or (super adj saturated)) with water with oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:24
S45	28302	(growth or yield) with plants and (method or process).clm.	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:29
S46	47	(growth or yield) with plants and (method or process).clm. and (saturated or supersaturated) near2 oxygen	US-PGPUB; USPAT; USOCR	OR	ON	2005/11/21 15:30
S47	140	(water adj electroly\$4) and anode same (platinum with iridium with oxide)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 18:13
S48	146	(water adj electroly\$4) and anode same (platinum with iridium with (oxide or dioxide))	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 18:53
S49	3	"3775284".pn.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 18:33
S50	4	"4100049".pn.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 18:47
S51	79	anode same (platinum with iridium with (oxide or dioxide)) same support	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2005/11/21 18:54

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

### CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 628 of 1320



### <u>326</u> <u>IN THE UNITED STATES PATENT AND TRADEMARK OFFICE</u>

In re Application of: James Andrew Senkiw Serial Number: 10/732,326 Title: Flow-Through Oxygenator Filing Date: December 10, 2003

Examiner: Not assigned Group art unit: Not assigned Att'y No.: AQI.002US1

MS PATENT APPLICATION Assistant Commissioner for Patents Box 1450 Alexandria, VA, 22313-1450

In compliance with the duty imposed by 37 C.F.R.§1.50 and in accordance with C.F.R.§§ 1.97 *et seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached form 1449 be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant further requests that a copy of the 1449 form, initialed by the Examiner to indicate that all listed citations have been considered, be returned with the next official communication.

Under C.F.R.§1,97 (b)(3), it is believed that no fee or certificate is required with this Information Disclosure Statement. If an official action has been mailed, the required fee will be paid. The Examiner is invited to contact the Applicant's representative at the below listed telephone number or e-mail address if there are any questions regarding this communication.

Respectfully submitted,

James Andrew Senkiw By his representative,

15 July 2004 Kathleen R. Terry Reg. No. 31884

2417 Como Avenue St. Paul, MN 55108-1459 651 659 9819 <u>Krterry@visi.com</u>

I hereby certify that these papers are being deposited with the USPS Service with sufficient first class postage and addressed to MS Patent Application, Commissioner for Patents, Box 1450, Alexandria, VA 22313-1450 on the date noted above.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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			<sup>US-</sup> 6,394,429				- Colvo				
			<sup>US-</sup> 6,315,886B1	11-13-1	2001	Zappi	et al.			······································	
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"EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. <sup>1</sup>Applicant's unique citation designation number (optional). <sup>2</sup> See Kinds Codes of USPTO Patent Documents at <u>www.uspto.gov</u> or MPEP 901.04. <sup>3</sup> Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). <sup>4</sup> For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. <sup>6</sup>Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. <sup>6</sup>Applicant is to place a check mark here if English language

Translation is attached. This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. If you need assistance in complete this form and/or the form cell 1.800-BTO, 9109 (1.800, 256, 9109) and solicat patien 2.

If you need assistance in completing the form, call 1-800-PTO-9199 (1-800-786-9199) and select option 2.

## CASE 0:20-cv-00358-ECT-HB Doc. 74 Filed 06/09/21 Page 630 of 1320

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## FLOW-THROUGH OXYGENATOR

#### **RELATED APPLICATIONS**

5 This application is a continuation-in-part of United States Patent Application Number 10/872,017, now United States Patent Number 6,xxx,xxx, which claims priority to United States Provisional Patent Application Number 60/358,534, filed February 22, 2002.

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#### FIELD OF THE INVENTION

This invention relates to the electrolytic generation of microbubbles of oxygen for increasing the oxygen content of flowing water. This invention also relates to the use of superoxygenated water to enhance the growth and yield of plants. The flow-through model is useful for oxygenating water for hydroponic plant culture, drip irrigation and waste water treatment.

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## **BACKGROUND OF THE INVENTION**

Many benefits may be obtained through raising the oxygen content of aqueous media. Efforts have been made to achieve higher saturated or supersaturated oxygen levels for applications such
as the improvement of water quality in ponds, lakes, marshes and reservoirs, the detoxification of contaminated water, culture of fish, shrimp and other aquatic animals, biological culture and hydroponic culture. For example, fish held in a limited environment such as an aquarium, a bait bucket or a live hold tank may quickly use up the dissolved oxygen in the course of normal respiration and are then subject to hypoxic stress, which can lead to death. A similar effect is
seen in cell cultures, where the respiring cells would benefit from higher oxygen content of the medium. Organic pollutants from agricultural, municipal and industrial facilities spread through the ground and surface water and adversely affect life forms. Many pollutants are toxic, carcinogenic or mutagenic. Decomposition of these pollutants is facilitated by oxygen, both by direct chemical detoxifying reactions or by stimulating the growth of detoxifying microflora.

30 Contaminated water is described as having an increased biological oxygen demand (BOD) and water treatment is aimed at decreasing the BOD so as to make more oxygen available for fish and other life forms.

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The most common method of increasing the oxygen content of a medium is by sparging with air or oxygen. While this is a simple method, the resulting large bubbles produced simply break the surface and are discharged into the atmosphere. Attempts have been made to reduce the size of

- 5 the bubbles in order to facilitate oxygen transfer by increasing the total surface area of the oxygen bubbles. United States Patent Number 5,534,143 discloses a microbubble generator that achieves a bubble size of about 0.10 millimeters to about 3 millimeters in diameter. United States Patent Number 6,394,429 ("the '429 patent") discloses a device for producing microbubbles, ranging in size from 0.1 to 100 microns in diameter, by forcing air into the fluid at
- 10 high pressure through a small orifice.

When the object of generating bubbles is to oxygenate the water, either air, with an oxygen content of about 21%, or pure oxygen may be used. The production of oxygen and hydrogen by the electrolysis of water is well known. A current is applied across an anode and a cathode

15 which are immersed in an aqueous medium. The current may be a direct current from a battery or an AC/DC converter from a line. Hydrogen gas is produced at the cathode and oxygen gas is produced at the anode. The reactions are:

AT THE CATHODE:	$4H_2O + 4e^- \rightarrow 4OH^- + 2H_2$
AT THE ANODE:	$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$
NET REACTION:	$6H_2O \rightarrow 4OH^- + 4H^+ + 2H_2 + O_2$

286 kilojoules of energy is required to generate one mole of oxygen.

The gasses form bubbles which rise to the surface of the fluid and may be collected. Either the oxygen or the hydrogen may be collected for various uses. The "electrolytic water" surrounding the anode becomes acidic while the electrolytic water surrounding the cathode becomes basic. 25 Therefore, the electrodes tend to foul or pit and have a limited life in these corrosive environments.

Many cathodes and anodes are commercially available. United States Patent Number 5,982,609 30 discloses cathodes comprising a metal or metallic oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten,

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**OWT Ex. 2118 Tennant Company v. OWT** IPR2021-00625

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manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium. Anodes are formed from the same metallic oxides or metals as cathodes. Electrodes may also be formed from alloys of the above metals or metals and oxides co-deposited on a substrate. The cathode and anodes may be formed on any convenient support in any desired shape or size. It is possible

- 5 to use the same materials or different materials for both electrodes. The choice is determined according to the uses. Platinum and iron alloys ("stainless steel") are often preferred materials due to their inherent resistance to the corrosive electrolytic water. An especially preferred anode disclosed in U. S. Patent Number 4,252,856 comprises vacuum deposited iridium oxide.
- 10 Holding vessels for live animals generally have a high population of animals which use up the available oxygen rapidly. Pumps to supply oxygen have high power requirements and the noise and bubbling may further stress the animals. The available electrolytic generators likewise have high power requirements and additionally run at high voltages and produce acidic and basic water which are detrimental to live animals. Many of the uses of oxygenators, such as keeping
- bait or caught fish alive, would benefit from portable devices that did not require a source of high power. The need remains for quiet, portable, low voltage means to oxygenate water.

It has also been known that plant roots are healthier when oxygenated water is applied. It is thought that oxygen inhibits the growth of deleterious fungi. The water sparged with air as in the '429 patent was shown to increase the biomass of hydroponically grown cucumbers and tomatoes by about 15%.

The need remains for oxygenator models suitable to be placed in-line in water distribution devices so as to be applied to field as well as hydroponic culture.

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## **SUMMARY OF THE INVENTION**

This invention provides an oxygen emitter which is an electrolytic cell which generates very small microbubbles and nanobubbles of oxygen in an aqueous medium, which bubbles are too small to break the surface tension of the medium, resulting in a medium supersaturated with oxygen.

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The electrodes may be a metal or oxide of at least one metal selected from the group consisting of ruthenium, iridium, nickel, iron, rhodium, rhenium, cobalt, tungsten, manganese, tantalum, molybdenum, lead, titanium, platinum, palladium and osmium or oxides thereof. The electrodes may be formed into open grids or may be closed surfaces. The most preferred cathode is a stainless steel mesh. The most preferred mesh is a 1/16 inch grid. The most preferred anode is platinum and iridium oxide on a support. A preferred support is titanium.

In order to form microbubbles and nanobubbles, the anode and cathode are separated by a critical distance. The critical distance ranges from 0.005 inches to 0.140 inches. The preferred critical distance is from 0.045 to 0.060 inches.

Models of different size are provided to be applicable to various volumes of aqueous medium to be oxygenated. The public is directed to choose the applicable model based on volume and power requirements of projected use. Those models with low voltage requirements are especially suited to oxygenating water in which animals are to be held.

Controls are provided to regulate the current and timing of electrolysis.

- A flow-through model is provided which may be connected in-line to a watering hose or to a hydroponic circulating system. The flow-through model can be formed into a tube with triangular cross-section. In this model, the anode is placed toward the outside of the tube and the cathode is placed on the inside, contacting the water flow. Alternatively, the anodes and cathodes may be in plates parallel to the long axis of the tube, or may be plates in a wafer stack.
- 25 Alternately, the electrodes may be placed in a side tube ("T" model) out of the direct flow of water. Protocols are provided to produce superoxygenated water at the desired flow rate and at the desired power usage. Controls are inserted to activate electrolysis when water is flowing and deactivate electrolysis at rest.
- 30 This invention includes a method to promote growth and increase yield of plants by application of superoxygenated water. The water treated with the emitter of this invention is one example of

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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superoxygenated water. Plants may be grown in hydroponic culture or in soil. The use of the flow-through model for drip irrigation of crops and waste water treatment is disclosed.

# **DESCRIPTION OF THE DRAWINGS**

Figure 1 is the  $O_2$  emitter of the invention.

Figure 2 is an assembled device.

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Figure 3 is a diagram of the electronic controls of the  $O_2$  emitter.

Figure 4 shows a funnel or pyramid variation of the O<sub>2</sub> emitter.

15 Figure 5 shows a multilayer sandwich  $O_2$  emitter.

Figure 6 shows the yield of tomato plants watered with superoxygenated water.

Figure 7 shows an oxygenation chamber suitable for flow-through applications. Figure 7A is a

20 cross section showing arrangement of three plate electrodes. Figure 7B is a longitudinal section showing the points of connection to the power source.

Figure 8 is a graph showing the oxygenation of waste water.

## 25 DETAILED DESCRIPTION OF THE INVENTION

Definitions:

For the purpose of describing the present invention, the following terms have these meanings:

30 "Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

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"Critical distance" means the distance separating the anode and cathode at which evolved oxygen forms microbubbles and nanobubbles.

"O<sub>2</sub> emitter" means a cell comprised of at least one anode and at least one cathode separated by the critical distance.

"Metal" means a metal or an alloy of one or more metals.

"Microbubble" means a bubble with a diameter less than 50 microns.

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"Nanobubble" means a bubble with a diameter less than that necessary to break the surface tension of water. Nanobubbles remain suspended in the water, giving the water an opalescent or milky appearance.

15 "Supersaturated" means oxygen at a higher concentration than normal calculated oxygen solubility at a particular temperature and pressure.

"Superoxygenated water" means water with an oxygen content at least 120% of that calculated to be saturated at a temperature.

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"Water" means any aqueous medium with resistance less than one ohm per square centimeter; that is, a medium that can support the electrolysis of water. In general, the lower limit of resistance for a medium that can support electrolysis is water containing more than 2000 ppm total dissolved solids.

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The present invention produces microbubbles and nanobubbles of oxygen via the electrolysis of water. As molecular oxygen radical (atomic weight 8) is produced, it reacts to form molecular oxygen,  $O_2$ . In the special dimensions of the invention, as explained in more detail in the following examples,  $O_2$  forms bubbles which are too small to break the surface tension of the

30 fluid. These bubbles remain suspended indefinitely in the fluid and, when allowed to build up, make the fluid opalescent or milky. Only after several hours do the bubbles begin to coalesce on

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the sides of the container and the water clears. During that time, the water is supersaturated with oxygen. In contrast, the  $H_2$  formed readily coalesces into larger bubbles which are discharged into the atmosphere, as can be seen by bubble formation at the cathode.

- 5 The first objective of this invention was to make an oxygen emitter with low power demands, low voltage and low current for use with live animals. For that reason, a small button emitter was devised. The anode and cathode were set at varying distances. It was found that electrolysis took place at very short distances before arcing of the current occurred. Surprisingly, at slightly larger distances, the water became milky and no bubbles formed at the anode, while hydrogen
- 10 continued to be bubbled off the cathode. At distance of 0.140 inches between the anode and cathode, it was observed that the oxygen formed bubbles at the anode. Therefore, the critical distance for microbubble and nanobubble formation was determined to be between 0.005 inches and 0.140 inches.

# 15 Example 1. Oxygen emitter.

As shown in Figure 1, the oxygen evolving anode 1 selected as the most efficient is an iridium oxide coated single sided sheet of platinum on a support of titanium (Eltech, Fairport Harbor, OH). The cathode 2 is a 1/16 inch mesh (size 8 mesh) marine stainless steel screen. The anode and cathode are separated by a non-conducting spacer 3 containing a gap 4 for the passage

- 20 of gas and mixing of anodic and cathodic water and connected to a power source through a connection point 5. Figure 2 shows a plan view of the assembled device. The O<sub>2</sub> emitter 6 with the anode connecting wire 7 and the cathode connecting wire 8 is contained in an enclosure 9, connected to the battery compartment 10. The spacer thickness is critical as it sets the critical distance. It must be of sufficient thickness to prevent arcing of the current, but thin enough to
- 25 separate the electrodes by no more than 0.140 inches. Above that thickness, the power needs are higher and the oxygen bubbles formed at higher voltage will coalesce and escape the fluid. Preferably, the spacer is from 0.005 to 0.075 inches thick. At the lower limits, the emitter tends to foul more quickly. Most preferably, the spacer is about 0.050 inches thick. The spacer may be any nonconductive material such as nylon, fiberglass, Teflon® polymer or other plastic.
- 30 Because of the criticality of the space distance, it is preferable to have a non-compressible spacer. It was found that Buna, with a durometer measure of 60 was not acceptable due to

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decomposition. Viton, a common fluoroelastomer, has a durometer measure of 90 and was found to hold its shape well.

In operation, a small device with an  $O_2$  emitter 1.485 inches in diameter was driven by 4AA batteries. The critical distance was held at 0.050 inches with a Viton spacer. Five gallons of water became saturated in seven minutes. This size is suitable for raising oxygen levels in an aquarium or bait bucket.

It is convenient to attach a control circuit which comprises a timer that is thermostatically controlled by a temperature sensor which determines the off time for the cathode. When the temperature of the solution changes, the resistance of the thermistor changes, which causes an off time of a certain duration. In cool water, the duration is longer so in a given volume, the emitter generates less oxygen. When the water is warmer and therefore hold less oxygen, the duration of off time is shorter. Thus the device is self-controlled to use power most

economically. Figure 3 shows a block diagram of a timer control with anode 1, cathode 2, thermistor temperature sensor 3, timer control circuit 4 and wire from a direct current power source 5.

#### Example 2. Measurement of O<sub>2</sub> bubbles.

- Attempts were made to measure the diameter of the O<sub>2</sub> bubbles emitted by the device of Example 1. In the case of particles other than gasses, measurements can easily be made by scanning electron microscopy, but gasses do not survive electron microscopy. Large bubble may be measured by pore exclusion, for example, which is also not feasible when measuring a gas bubble. A black and white digital, high contrast, backlit photograph of treated water with a millimeter scale reference was shot of water produced by the emitter of Example 1. About 125 bubbles were seen in the area selected for measurement. Seven bubbles ranging from the smallest clearly seen to the largest were measured. The area was enlarged, giving a scale multiplier of 0.029412.
- 30 Recorded bubble diameters at scale were 0.16, 0.22, 0.35, 0.51, 0.76, 0.88 and 1.09 millimeters. The last three were considered outliers by reverse analysis of variance and were assumed to be

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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hydrogen bubbles. When multiplied by the scale multiplier, the assumed  $O_2$  bubbles were found to range from 4.7 to 15 microns in diameter. This test was limited by the resolution of the camera and smaller bubbles in the nanometer range could not be resolved. It is known that white light cannot resolve features in the nanometer size range, so monochromatic laser light may give resolution sensitive enough to measure smaller bubbles. Efforts continue to increase the

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sensitivity of measurement so that sub-micron diameter bubbles can be measured.

## Example 3. Other models of oxygen emitter

Depending on the volume of fluid to be oxygenated, the oxygen emitter of this invention may be shaped as a circle, rectangle, cone or other model. One or more may be set in a substrate that may be metal, glass, plastic or other material. The substrate is not critical as long as the current is isolated to the electrodes by the nonconductor spacer material of a thickness from 0.005 to 0.075 inches, preferably 0.050 inches. It has been noticed that the flow of water seems to be at the periphery of the emitter, while the evolved visible bubbles (H<sub>2</sub>) arise at the center of the

15 emitter. Therefore, a funnel or pyramidal shaped emitter was constructed to treat larger volumes of fluid. Figure 4 is a cross sectional diagram of such an emitter. The anode 1 is formed as an open grid separated from a marine grade stainless steel screen cathode 2 by the critical distance by spacer 3 around the periphery of the emitter and at the apex. This flow-through embodiment is suitable for treating large volumes of water rapidly.

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The size may be varied as required. A round emitter for oxygenating a bait bucket may be about 2 inches in diameter, while a 3-inch diameter emitter is adequate for oxygenating a 10 to 40 gallon tank. The live well of a fishing boat will generally hold 40 to 80 gallons of water and require a 4-inch diameter emitter. It is within the scope of this invention to construct larger emitters or to use several in a series to oxygenate larger volumes. It is also within the scope of this invention to vary the model to provide for low voltage and amperage in cases where the need for oxygen is moderate and long lasting or conversely, to supersaturate water very quickly at higher voltage and amperage. In the special dimensions of the present invention, it has been found that a 6 volt battery supplying a current as low as 40 milliamperes is sufficient to generate

30 oxygen. Such a model is especially useful with live plants or animals, while it is more convenient for industrial use to use a higher voltage and current. Table I shows a number of

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

models suitable to various uses.

Emitter Model	Gallons	Volts	Amps Max	k. Ave	Watts
Bait keeper	5	6	0.090	0.060	0.36
Livewell	32	12	0.180	0.120	1.44
OEM 2 inch	10	12	0.210	0.120	1.44
Bait store	70	12	0.180	0.180	2.16
Double cycle	2	12	0.180	0.180	2.16
OEM 3 inch	50	12	0.500	0.265	3.48
OEM 4 inch	80	12	0.980	0.410	4.92
Water pail	2	24	1.200	1.200	28.80
Plate	250	12	5.000	2.500	30.00

### TABLE I

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# 15 Example 4. Multilayer sandwich O<sub>2</sub> emitter

An O<sub>2</sub> emitter was made in a multilayer sandwich embodiment. (Figure 5) An iridium oxide coated platinum anode 1 was formed into a grid to allow good water flow and sandwiched between two stainless steel screen cathodes 2. Spacing was held at the critical distance by nylon spacers 3. The embodiment illustrated is held in a cassette 4 which is secured by nylon bolt 5

20 with a nylon washer 6. The dimensions selected were:

cathode screen	0.045 inches thick
nylon spacer	0.053 inches thick
anode grid	0.035 inches thick
nylon spacer	0.053 inches thick
cathode screen	0.045 inches thick,

for an overall emitter thickness of 0.231 inches.

If a more powerful emitter is desired, it is within the scope of this invention to repeat the sequence of stacking. For example, an embodiment may easily be constructed with this

30 sequence: cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode, spacer, anode, spacer, cathode. The number of layers in the sandwich is limited only by the power requirements acceptable for an application.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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# Example 5. Effect of superoxygenated water on the growth of plants.

It is known that oxygen is important for the growth of plants. Although plants evolve oxygen during photosynthesis, they also have a requirement for oxygen for respiration. Oxygen is

- 5 evolved in the leaves of the plants, while often the roots are in a hypoxic environment without enough oxygen to support optimum respiration, which can be reflected in less than optimum growth and nutrient utilization. Hydroponically grown plants are particularly susceptible to oxygen deficit in the root system. United States Patent Number 5,887,383 describes a liquid supply pump unit for hydroponic cultures which attain oxygen enrichment by sparging with air.
- 10 Such a method has high energy requirements and is noisy. Furthermore, while suitable for selfcontained hydroponic culture, the apparatus is not usable for field irrigation. In a report available on the web, it was shown that hydroponically grown cucumbers and tomatoes supplied with water oxygenated with a device similar to that described in the '429 patent had increased biomass of about 12% and 17% respectively. It should be noted that when sparged with air, the water may

become saturated with oxygen, but it is unlikely that the water is superoxygenated.

# A. Superoxygenated water in hydroponic culture.

Two small hydroponic systems were set up to grow two tomato plants. Circulation protocols were identical except that the 2 ½ gallon water reservoir for the Control plant was eroated with
and aquarium bubbler and that for the Test plant was oxygenated with a five-inch strip emitter for two minutes prior to pumping. The cycle was set at four minutes of pumping, followed by four minutes of rest. The control water had an oxygen content of about 97% to 103% saturation, that is, it was saturated with oxygen. The test water had an oxygen content of about 153% to165% saturation, that is, it was supersaturated. The test plant was at least four times the volume of the control plant and began to show what looked like fertilizer burn. At that point the fertilizer for the Test plant was reduced by half. Since the plants were not exposed to natural light but to continuous artificial light in an indoor environment without the natural means of fertilization (wind and/or insects), the experiment was discontinued after three months. At that time, the Test plant but not the Control plant had blossomed.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

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## B. Superoxygenated water in field culture.

A pilot study was designed to ascertain that plants outside the hydroponic culture facility would benefit from the application of oxygen. It was decided to use water treated with the emitter of Example 1 as the oxygen carrier. Since water so treated is supersaturated, it is an excellent carrier of oxygen

5 of oxygen.

Tomato seeds (Burpee "Big Boy") were planted in one-inch diameter peat and dirt plugs encased in cheese cloth and placed in a tray in a southwest window. Controls were watered once a day with tap water ("Control") or oxygenated water ("Test"). Both Controls and Test sprouted at one

- 10 week. After five weeks, the Test plants were an average of 11 inches tall while the Controls were an average of nine inches tall. At this time, May 10, when the threat of frost in Minnesota was minimal, the plants were transplanted to 13 inch diameter pots with drainage holes. Four inches of top soil was added to each pot, topped off with four inches of Scott's Potting Soil. The pots were placed outside in a sunny area with at least eight hours a day of full sun. The plants were
- 15 watered as needed with either plain tap water (Control) or oxygenated water (Test). The oxygenated water was produced by use of the emitter of Example 1 run for one-half hour in a five-gallon container of water. Previous experiments showed that water thus treated had an oxygen content from 160% to 260% saturation. The Test plants flowered on June 4, while the Controls did not flower until June 18. For both groups, every plant in the group first had flowers
- 20 on the same day. All plants were fertilized on July 2 and a soaker hose provided because the plants were now so big that watering by hand was difficult. The soaker hose was run for one half to one hour each morning, depending on the weather, to a point at which the soil was saturated with water. One half hour after the soaker hose was turned off, about 750 ml of superoxygenated water was applied to each of the Test plants.

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The Test plants were bushier than the Controls although the heights were similar. At this time, there were eight Control plants and seven Test plants because one of the Test plants broke in a storm. On July 2, the control plants averaged about 17 primary branches from the vine stem, while the control plants averaged about 13 primary branches from the vine stem. As the

30 tomatoes matured, each was weighed on a kitchen scale at harvest. The yield history is shown in Table II.

OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

# TABLE II

Week of:	Control,	grams tomatoes from	Test, grams tomatoes from			
	eight plar	eight plants/cumulative total		seven plants/cumulative tot		
July 27	240		400			
August 3	180	420	2910	3310		
August 10	· 905	1325	1830	5140		
August 17	.410	1735	2590	7730		
August 24	3300	5035	2470	10200		
August 31	4150	9175	1580	11780		
September 15	not weighed	1	3710	15490		
Final Harvest September 24	6435	15620	8895	24385		

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The total yield for the eight Control plants was 15620 grams or 1952 grams of tomatoes per plant.

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The total yield for the seven Test plants was 24385 grams or 3484 grams of tomatoes per plant, an increase in yield of about 79% over the Control plants.

Figure 6 shows the cumulative total as plotted against time. Not only did the Test plants blossom and bear fruit earlier, but that the Control plants never caught up to the test plants in the short Minnesota growing season. It should be noted that the experiment was terminated because of predicted frost. All fruits, both green and red, were harvested and weighed at that point.

## Example 6. Flow-through emitter for agricultural use.

In order to apply the findings of example 5 to agricultural uses, an emitter than can oxygenate running water efficiently was developed. In Figure 7 (A), the oxygenation chamber is

25 comprised of three anodes 1 and cathodes 2, of appropriate size to fit inside a tube or hose and separated by the critical distance are placed within a tube or hose 3 at 120° angles to each other. The anodes and cathodes are positioned with stabilizing hardware 4. The stabilizing hardware, which can be any configuration such as a screw, rod or washer, is preferably formed from stainless steel. Figure 7 (B) shows a plan view of the oxygenation chamber with stabilizing

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hardware 4 serving as a connector to the power source and stabilizing hardware 5 serving as a connector to the power source. The active area is shown at 6.

This invention is not limited to the design selected for this embodiment. Those skilled in the art

- 5 can readily fabricate any of the emitters shown in Figures 4 or 5, or can design other embodiments that will oxygenate flowing water. One useful embodiment is the "T" model, wherein the emitter unit is set in a side arm. The emitted bubbles are swept into the water flow. The unit is detachable for easy servicing. Table III shows several models of flow through emitters. The voltage and flowrates were held constant and the current varied. The Dissolved
- 10 oxygen (DO) from the source was 7.1 mg/liter. The starting temperature was 12.2° C but the flowing water cooled slightly to 11 or 11.5° C. Without undue experimentation, anyone may easily select the embodiment that best suits desired characteristics from Table III or designed with the teachings of Table III.

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## TABLE III

MODEL	ACTIVE	VOLTAGE	CURRENT,	FLOW RATE	DO OF*
	ELECTRODE		AMPS	GAL/MINUTE	SAMPLE AT
	AREA, SQ.IN.				ONE MINUTE
2-inch "T"	2	28.3	0.7	12	N/A
3-inch "T"	3	28.3	1.75	12	N/A
2-plate Tube	20	28.3	9.1	12	8.4
3-Plate tube	30	28.3	12.8	12	9.6

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\*As the apparatus runs longer, the flowing water becomes milky, indicating supersaturation. The one-minute time point shows the rapid increase in oxygenation.

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The following plants will be tested for response to superoxygenated water: grape vines, lettuce, and radishes in three different climate zones. The operators for these facilities will be supplied with units for drip irrigation. Drip irrigation is a technique wherein water is pumped through a pipe or hose with perforations at the site of each plant to be irrigated. The conduit may be underground or above ground. Since the water is applied directly to the plant rather than wetting

the entire field, this technique is especially useful in arid climates or for plants requiring high fertilizer applications.

The superoxygenated water will be applied by drip irrigation per the usual protocol for the respective plants. Growth and yield will be compared to the same plants given only the usual irrigation water. Pest control and fertilization will be the same between test and control plants, except that the operators of the experiments will be cautioned to be aware of the possibility of fertilizer burn in the test plants and to adjust their protocols accordingly.

10 It is expected that the superoxygenated plants with drip irrigation will show more improved performance with more continuous application of oxygen than did the tomato plants of Example 5, which were given superoxygenated water only once a day.

# Example 7. Treatment of waste water.

15 Waste water, with a high organic content, has a high BOD, due to the bacterial flora. It is desirable to raise the oxygen content of the waste water in order to cause the flora to flocculate. However, it is very difficult to effectively oxygenate such water. Using a 4 inch OEM (see Table I) with a 12 volt battery, four liters of waste water in a five gallon pail were oxygenated. As shown in Figure 8, the dissolved oxygen went from 0.5 mg/l to 10.8 mg/l in nine minutes.

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Those skilled in the art will readily comprehend that variations, modifications and additions may in the embodiments described herein may be made. Therefore, such variations, modifications and additions are within the scope of the appended claims.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

I claim:

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Claim 1. A flow-through oxygenator comprising an emitter for electrolytic generation of microbubbles of oxygen comprising an anode separated at a critical distance from a cathode and a power source all in electrical communication with each other, wherein the emitter is placed within or adjacent to a conduit for flowing water.

Claim 2. The emitter of claim 1 wherein the anode is a metal or a metallic oxide or a combination of a metal and a metallic oxide and the anode is platinum and iridium oxide on a support and the cathode is a metal or metallic oxide or a combination of a metal and a metallic oxide.

Claim 3. The critical distance of claim 1 which is 0.005 to 0.140 inches.

15 Claim 4. The critical distance of claim 1 which is 0.045 to 0.060 inches.

Claim 5. The product of claim 1 wherein the water is supersaturated with oxygen and of an approximately neutral pH.

20 Claim 6. A method for enhancing growth and yield of plants comprising the administration of supersaturated water on said plants.

Claim 7. The method of claim 6 wherein the supersaturated water is delivered to the plants in hydroponic culture or through drip irrigation.

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Claim 8. A method for treating waste water comprising passing the waste water through a conduit comprising the emitter of claim 1.

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OWT Ex. 2118 Tennant Company v. OWT IPR2021-00625

JA646

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## ABSTRACT

An oxygen emitter which is an electrolytic cell is disclosed. When the anode and cathode are separated by a critical distance, very small microbubbles and nanobubbles of oxygen are

5 generated. The very small oxygen bubbles remain in suspension, forming a solution supersaturated in oxygen. A flow-through model for oxygenating flowing water is disclosed. The use of supersaturated water for enhancing the growth of plants is disclosed. Methods for applying supersaturated water to plants manually, by drip irrigation or in hydroponic culture are described. The treatment of waste water by raising the dissolved oxygen with the use of an

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10 oxygen emitter is disclosed.

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